

AN 122:122242 CA
 TI Determination of enantiomeric composition of 2-phenyl-2-(2-piperidyl)acetamide. A routine method for evaluation of enantiomeric purity of primary amides
 AU **Jursic, Branko S.**; Zdravkovski, Zoran; **Zuanic, Miljenko**
 CS Dep. Chem., Univ. New Orleans, New Orleans, LA, 70148, USA
 SO Tetrahedron: Asymmetry (1994), 5(9), 1711-16
 CODEN: TASYE3; ISSN: 0957-4166
 DT Journal
 LA English
 AB Several NMR chiral resolving agents have successfully been used to demonstrate their usefulness for the detn. of the enantiomeric compn. of the title compd.

=> select

ENTER ANSWER SET OR SMARTSELECT L# OR (L5):15

ENTER ANSWER NUMBER OR RANGE (1-):1

ENTER DISPLAY CODE (TI) OR ?:rn

E1 THROUGH E10 ASSIGNED

=> fil reg

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	ENTRY	SESSION
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 DICTIONARY FILE UPDATES: 16 JUL 2000 HIGHEST RN 277740-73-5

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 11, 2000

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 conducting SmartSELECT searches.

Structure search limits have been increased. See HELP SLIMIT
 for details.

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 (101401-81-4/RN)
 1 150723-37-8/BI

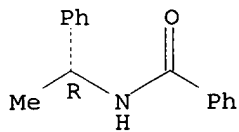
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 1 20826-48-6/BI
 (20826-48-6/RN)
 1 69632-32-2/BI
 (69632-32-2/RN)

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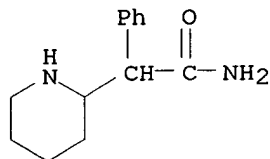
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN Benzamide, N-[(1R)-1-phenylethyl]- (9CI)
 MF C15 H15 N O

Absolute stereochemistry.



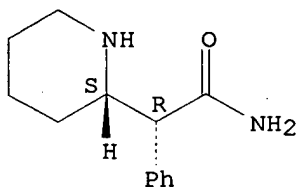
HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):9

L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN 2-Piperidineacetamide, .alpha.-phenyl- (6CI, 7CI, 8CI, 9CI)
 MF C13 H18 N2 O
 CI COM



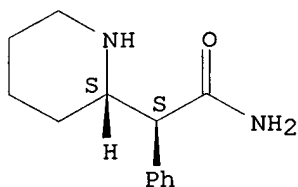
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN 2-Piperidineacetamide, .alpha.-phenyl-, (.alpha.R,2S)- (9CI)
 MF C13 H18 N2 O

Absolute stereochemistry. Rotation (+).



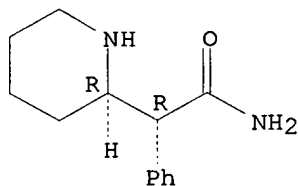
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN 2-Piperidineacetamide, .alpha.-phenyl-, (.alpha.S,2S)- (9CI)
 MF C13 H18 N2 O

Absolute stereochemistry. Rotation (-).



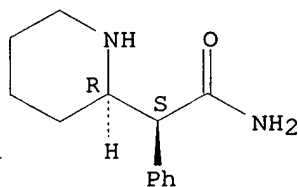
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN 2-Piperidineacetamide, .alpha.-phenyl-, (.alpha.R,2R)- (9CI)
 MF C13 H18 N2 O

Absolute stereochemistry. Rotation (+).



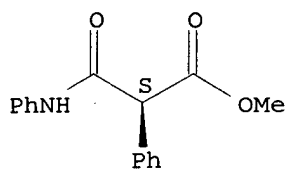
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN 2-Piperidineacetamide, .alpha.-phenyl-, (.alpha.S,2R)- (9CI)
 MF C13 H18 N2 O

Absolute stereochemistry. Rotation (-).



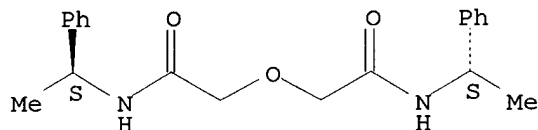
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN Benzeneacetic acid, .alpha.-[(phenylamino)carbonyl]-, methyl ester, (S)- (9CI)
 MF C16 H15 N O3

Absolute stereochemistry.



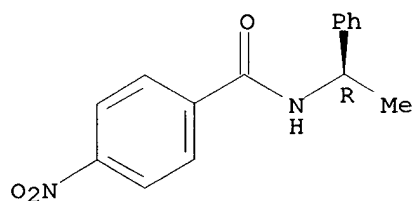
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN Acetamide, 2,2'-oxybis[N-(1-phenylethyl)-, [S-(R*,R*)]]- (9CI)
 MF C20 H24 N2 O3

Absolute stereochemistry.



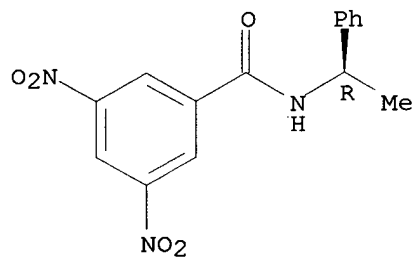
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN Benzamide, 4-nitro-N-(1-phenylethyl)-, (R)- (9CI)
 MF C15 H14 N2 O3

Absolute stereochemistry.



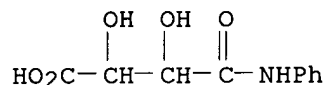
L6 10 ANSWERS REGISTRY COPYRIGHT 2000 ACS
 IN Benzamide, 3,5-dinitro-N-[(1R)-1-phenylethyl]- (9CI)
 MF C15 H13 N3 O5
 CI COM

Absolute stereochemistry. Rotation (-).



ALL ANSWERS HAVE BEEN SCANNED

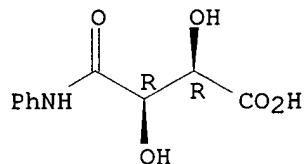
RN 6051-31-6 REGISTRY
 CN Butanoic acid, 2,3-dihydroxy-4-oxo-4-(phenylamino)- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN **Tartranilic acid (7CI, 8CI)**
 FS 3D CONCORD
 MF C10 H11 N O5
 LC STN Files: BEILSTEIN*, CAOLD
 (*File contains numerically searchable property data)



1 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

L4 ANSWER 2 OF 2 REGISTRY COPYRIGHT 2001 ACS
 RN 3019-58-7 REGISTRY
 CN Butanoic acid, 2,3-dihydroxy-4-oxo-4-(phenylamino)-, (2R,3R)- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN Butanoic acid, 2,3-dihydroxy-4-oxo-4-(phenylamino)-, [R-(R*,R*)]-
 CN **Tartranilic acid (8CI)**
 OTHER NAMES:
 CN (R,R)-Tartranilic acid
 FS STEREOSEARCH
 MF C10 H11 N O5
 CI COM
 LC STN Files: BEILSTEIN*, CA, CAOLD, CAPLUS, CASREACT, CHEMCATS, CSCHEM
 (*File contains numerically searchable property data)

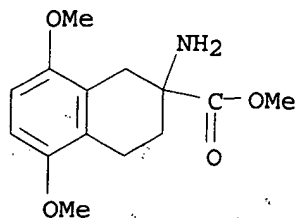
Absolute stereochemistry. Rotation (+).



3 REFERENCES IN FILE CA (1967 TO DATE)
 3 REFERENCES IN FILE CAPLUS (1967 TO DATE)
 1 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 129:244880
 REFERENCE 2: 129:216421
 REFERENCE 3: 106:101729

methyl ester, (.-.-)-
 FS 3D CONCORD
 MF C14 H19 N O4
 SR CA
 LC STN Files: CA, CAPLUS, CASREACT, TOXCENTER

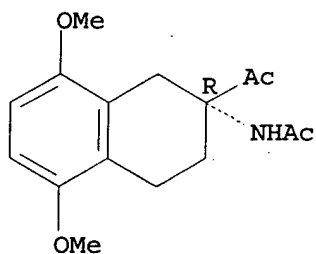


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

3 REFERENCES IN FILE CA (1967 TO DATE)
 3 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 7 OF 8 REGISTRY COPYRIGHT 2002 ACS
 RN 86264-61-1 REGISTRY
 CN Acetamide, N-(2-acetyl-1,2,3,4-tetrahydro-5,8-dimethoxy-2-naphthalenyl)-,
 (R)- (9CI) (CA INDEX NAME)
 FS STEREOSEARCH
 MF C16 H21 N O4
 LC STN Files: CA, CAPLUS, CASREACT, TOXCENTER, USPATFULL

Absolute stereochemistry.

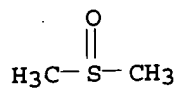


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

4 REFERENCES IN FILE CA (1967 TO DATE)
 4 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 8 OF 8 REGISTRY COPYRIGHT 2002 ACS
 RN 67-68-5 REGISTRY
 CN Methane, sulfinylbis- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN Methyl sulfoxide (8CI)
 OTHER NAMES:
 CN Demsodrox
 CN Dimethyl sulfoxide
 CN Dimethyl sulphoxide
 CN Dimexide
 CN Dimexidum
 CN Dipirartril-tropico
 CN DMS 70

CN DMS 90
 CN DMSO
 CN Dolicur
 CN Dromisol
 CN Durasorb
 CN Hyadur
 CN Infiltrina
 CN Somipront
 CN SQ 9453
 CN Sulfinylbismethane
 FS 3D CONCORD
 DR 8070-53-9, 164071-41-4
 MF C2 H6 O S
 CI COM
 LC STN Files: ADISNEWS, AGRICOLA, ANABSTR, BEILSTEIN*, BIOBUSINESS, BIOSIS,
 BIOTECHNO, CA, CABA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN,
 CHEMCATS, CHEMINFORMRX, CHEMLIST, CHEMSAFE, CIN, CSCHEM, CSNB, DDFU,
 DETHERM*, DIOGENES, DIPPR*, DRUGU, EMBASE, ENCOMPLIT, ENCOMPLIT2,
 ENCOMPPAT, ENCOMPPAT2, GMELIN*, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB,
 IPA, MEDLINE, MRCK*, MSDS-OHS, NAPRALERT, NIOSHTIC, PDLCOM*, PIRA,
 PROMT, RTECS*, SPECINFO, TOXCENTER, TULSA, ULIDAT, USAN, USPAT2,
 USPATFULL, VETU, VTB
 (*File contains numerically searchable property data)
 Other Sources: DSL**, EINECS**, TSCA**, WHO
 (**Enter CHEMLIST File for up-to-date regulatory information)



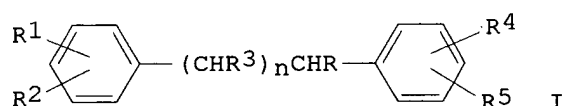
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

23731 REFERENCES IN FILE CA (1967 TO DATE)
 373 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
 23755 REFERENCES IN FILE CAPLUS (1967 TO DATE)
 39 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

AN 97:38738 CA
TI Enantiomeric .alpha.-aminopropiophenones (cathinone): preparation and investigation
AU Berrang, Bertold D.; Lewin, Anita H.; Carroll, F. Ivy
CS Chem. Life Sci. Group, Research Triangle Inst., Research Triangle Park, NC, 27709, USA
SO J. Org. Chem. (1982), 47(13), 2643-7
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
AB (-)-PhCOCHMeNH₂ is a constituent of *Catha edulis*. Resoln. of (.-.-)-PhCOCHMeNH₂ with mandelic acid gave only minute yields and tartaric acid gave only somewhat better results. (.-.-)-Norephedrine was resolved in high yield into its (+) and (-) antipodes with O,O-dibenzoyl-d-tartaric acid and each enantiomer was converted into its N-formyl deriv. and oxidized with CrO₃ in pyridine. Hydrolysis with 20% HCl at 40.degree. gave optically pure PhCOCHMeNH₂.HCl without racemization. (-)-PhCOCHMeNH₂ was obtained in 39% and the (+) enantiomer in 40% overall yield from (.-.-)-norephedrine. The characterization of PhCOCHMeNH₂ and its salts and their stability in various solvents were discussed.

AN 1986:148472 CAPLUS
 DN 104:148472
 TI Synthesis of amine derivatives
 IN Masuko, Fujio; Katsura, Tadashi
 PA Sumitomo Chemical Co., Ltd. , Japan
 SO U.S., 13 pp. Cont.-in-part of U.S. Ser. No. 65,429, abandoned.
 CODEN: USXXAM
 DT Patent
 LA English
 FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4536599	A	19850820	US 1979-90479	19791101
	JP 55028959	A2	19800229	JP 1978-102614	19780822
	JP 61055488	B4	19861128		
	JP 55033442	A2	19800308	JP 1978-106541	19780830
	JP 62000905	B4	19870110		
PRAI	JP 1978-102614		19780822		
	JP 1978-106541		19780830		
	US 1979-65429		19790810		
GI					



AB Diphenylalkylamines I (R = NH₂; R₁-R₅ = H, halo, OH, trihalomethyl, Ph, PhO, PhS, alkyl, alkenyl, alkoxy, alkylthio, dialkylamino, alkylsulfonyl; n = 2, 3), useful as pharmaceutical intermediates and optical resolu. **agents**, were prep'd. by condensing R₄R₅C₆H₃CH₂CN with R₁R₂C₆H₃(CHR₃)_nX (X = halo) in the presence of a base, hydrolysis of the resultant I (R = cyano) by H₂O₂ and a base in the presence of an org. quaternary ammonium salt, and Hofmann rearrangement of the resultant I (R = CONH₂) in the presence of a base. Thus, 4-ClC₆H₄CH₂CN, PhCH₂Cl, Bu₄NBr, and 25% aq. NaOH reacted in PhMe to give 95% PhCH₂CHRC₆H₄Cl-4 (II; R = cyano), which was hydrolyzed by aq. NaOH-H₂O₂ in MeOH in the presence of Bu₄NBr to give 98% II (R = CONH₂). Rearrangement of the **amide** by Br-NaOH in MeOH gave 97% II (R = NH₂) (III), which was **resolved** by L-(+)-**tartaric** acid to give 50% resolu. yield of 1-III.

L1 ANSWER 1 OF 1 CA COPYRIGHT 2000 ACS
 AN 122:122242 CA
 TI Determination of enantiomeric composition of 2-phenyl-2-(2-piperidyl)acetamide. A routine method for evaluation of enantiomeric purity of primary amides
 AU Jursic, Branko S.; Zdravkovski, Zoran; Zuanic, Miljenko
 CS Dep. Chem., Univ. New Orleans, New Orleans, LA, 70148, USA
 SO Tetrahedron: Asymmetry (1994), 5(9), 1711-16
 CODEN: TASYE3; ISSN: 0957-4166
 DT Journal
 LA English

=> select

ENTER ANSWER SET OR SMARTSELECT L# OR (L1):11

ENTER ANSWER NUMBER OR RANGE (1-):1

ENTER DISPLAY CODE (TI) OR ?:rn

E1 THROUGH E10 ASSIGNED

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COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	2.58	2.73

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 DICTIONARY FILE UPDATES: 16 JUL 2000 HIGHEST RN 277740-73-5

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 11, 2000

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Structure search limits have been increased. See HELP SLIMIT for details.

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 (101401-81-4/RN)
 1 150723-37-8/BI
 (150723-37-8/RN)
 1 160618-08-6/BI
 (160618-08-6/RN)
 1 160707-36-8/BI
 (160707-36-8/RN)
 1 160707-37-9/BI

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      (160707-37-9/RN)
1 160707-38-0/BI
      (160707-38-0/RN)
1 160707-39-1/BI
      (160707-39-1/RN)
1 19395-39-2/BI
      (19395-39-2/RN)
1 20826-48-6/BI
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1 69632-32-2/BI
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160707-36 -8/BI OR 160707-37-9/BI OR 160707-38-0/BI OR 160707-39-1/BI OR
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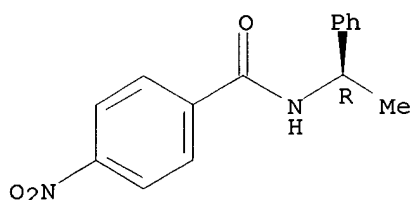
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AN 1993:512575 CAPLUS
DN 119:112575
TI Direct optical resolution of enantiomers by liquid chromatography using
(R,R)-tartramide derivative as a chiral selector
AU Tsuchihashi, Yasuo; Tsuchihashi, Akira
CS Tokyo Pharma Coll., Tokyo, Japan
SO Yakubutsu Dotai (1993), 8(1), 127-39
CODEN: YADOEL; ISSN: 0916-1139
DT Journal
LA Japanese
AB (R,R)-N,N'-Diisopropyltartramide (I) and its **analog** show
enantioselectivity toward many compds. possessing at least two hydrogen
bond sites. Addn. of I to the nonaq. mobile-phase solvent in silica gel
chromatog. as well as the use of a silica-based **chiral**
stationary phase derived from (R,R)-**tartramide** (II) thus make
possible the direct resoln. of a wide range of enantiomers. Such
enantiomers contain .alpha.-hydroxy carbonyl derivs., .beta.-hydroxy
carbonyl derivs., N-acyl derivs. of primary amines, amino esters, and
.beta.-amino alcs., glutarimides, barbiturates, .alpha.-hydroxy
ketoximes,
bi-.beta.-naphthol and 1,2-diols. The mode of complexation for the obsd.
enantioselection was dual hydrogen bonding between II derivs. and the
solute enantiomers to be resolved. This mode of hydrogen bonding leads
to
the formation of a pair of transient diastereomers differing in
stability.
The broad scope of application of the II deriv. as a **chiral**
selector may possibly be due to conformational changes involving the
formation and/or scission of intramol. hydrogen bond(s) at the time of
complexation. By such conformational reorganization of the II deriv.,
this mol. is able to provide dual hydrogen bond sites complementary to
those of the versatile enantiomers listed above. Dual hydrogen bonding
was obsd. in an x-ray crystal structure of the complex of I with
(S,S)-9,10-dimethyl-9,10-dihydrophenanthrene-9,10-diol. In the complex,
intermol. hydrogen bonds are formed between hydroxyls of the diol and
amide carbonyls of II. Dual hydrogen bonds in this complex are
characterized by relative orientation of the set of bonding sites of each
component showing a twist, rotational sense of which reflects the abs.
configuration.

L3 ANSWER 1 OF 3 REGISTRY COPYRIGHT 2000 ACS
 RN **101401-81-4** REGISTRY
 CN Benzamide, 4-nitro-N-(1-phenylethyl)-, (R)- (9CI) (CA INDEX NAME)
 FS STEREOSEARCH
 MF **C15 H14 N2 O3**
 SR CA
 LC STN Files: BEILSTEIN*, CA, CAPLUS, USPATFULL
 (*File contains numerically searchable property data)

Absolute stereochemistry.

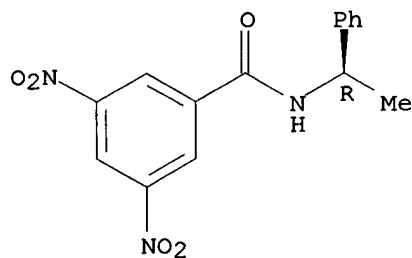


6 REFERENCES IN FILE CA (1967 TO DATE)
 6 REFERENCES IN FILE CAPLUS (1967 TO DATE)

=> d 13 2-3

L3 ANSWER 2 OF 3 REGISTRY COPYRIGHT 2000 ACS
 RN **69632-32-2** REGISTRY
 CN Benzamide, 3,5-dinitro-N-[(1R)-1-phenylethyl]- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN Benzamide, 3,5-dinitro-N-(1-phenylethyl)-, (R)-
 OTHER NAMES:
 CN (-)-N-3,5-Dinitrobenzoyl-.alpha.-methylbenzylamine
 CN (R)-(-)-N-(3,5-Dinitrobenzoyl)-.alpha.-methylbenzylamine
 CN (R)-N-(1-Phenethyl)-3,5-dinitrobenzamide
 CN (R)-N-(1-Phenylethyl)-3,5-dinitrobenzamide
 CN N-(3,5-Dinitrobenzoyl)(-)-1-phenylethylamine
 FS STEREOSEARCH
 MF **C15 H13 N3 O5**
 CI COM
 LC STN Files: BEILSTEIN*, CA, CAPLUS, CASREACT, CHEMCATS, CSCHEM,
 MSDS-OHS,
 TOXLIT, USPATFULL
 (*File contains numerically searchable property data)

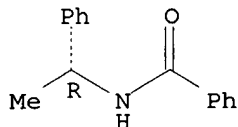
Absolute stereochemistry. Rotation (-).



58 REFERENCES IN FILE CA (1967 TO DATE)
 1 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
 59 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L3 ANSWER 3 OF 3 REGISTRY COPYRIGHT 2000 ACS
 RN **20826-48-6** REGISTRY
 CN Benzamide, N-[(1R)-1-phenylethyl]- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN Benzamide, N-(.alpha.-methylbenzyl)-, (+)- (8CI)
 CN Benzamide, N-(1-phenylethyl)-, (R)-
 OTHER NAMES:
 CN N-[(R)-1-Phenylethyl]benzamide
 FS STEREOSEARCH
 MF **C15 H15 N O**
 LC STN Files: BEILSTEIN*, CA, CAPLUS, CHEMINFORMRX, TOXLIT, USPATFULL
 (*File contains numerically searchable property data)

Absolute stereochemistry.



20 REFERENCES IN FILE CA (1967 TO DATE)
 20 REFERENCES IN FILE CAPLUS (1967 TO DATE)

=> s 13

L4 75 L3

=> s 14 and 9(camphor?(w)sulphon?) or malic or madelic or (bis(w)naphth?) or glucuronic)

MISSING OPERATOR 'AND 9(CAMPBOR?)'

The search profile that was entered contains terms or nested terms that are not separated by a logical operator.

=> s 14 and ((camphor?(w)sulphon?) or malic or madelic or (bis(w)naphth?) or glucuronic)

10884 CAMPBOR?
352 SULPHON?
0 CAMPBOR?(W)SULPHON?
16606 MALIC
2 MADELIC
274473 BIS
200317 NAPHTH?
392 BIS(W)NAPHTH?
6557 GLUCURONIC

L5 0 L4 AND ((CAMPBOR?(W)SULPHON?) OR MALIC OR MADELIC OR (BIS(W)NAPHTH?) OR GLUCURONIC)

=> s 14 and (camphorsulphon? or malic or madelic or (bis(w)naphth?) or glucuronic)

4 CAMPHORSULPHON?
16606 MALIC
2 MADELIC
274473 BIS
200317 NAPHTH?
392 BIS(W)NAPHTH?
6557 GLUCURONIC

L6 0 L4 AND (CAMPHORSULPHON? OR MALIC OR MADELIC OR (BIS(W)NAPHTH?) OR GLUCURONIC)

=> s 14 and (acid?(l)resolv?)

2931215 ACID?
123278 RESOLV?
13657 ACID?(L)RESOLV?
11 L4 AND (ACID?(L)RESOLV?)

L7

=> d bib abs 1-11

L7 ANSWER 1 OF 11 CAPLUS COPYRIGHT 2000 ACS
AN 1999:692221 CAPLUS
DN 132:8574
TI New chiral stationary phases based on (R)-1-naphthylethylamine bound to 2,4,5,6-tetrachloro-1,3-dicyanobenzene
AU Kontrec, Darko; Vinkovic, Vladimir; Sunjic, Vitomir

CS Ruder Boskovic Institute, Zagreb, HR-10000, Croatia
SO Chirality (1999), 11(9), 722-730
CODEN: CHRLEP; ISSN: 0899-0042
PB Wiley-Liss, Inc.
DT Journal
LA English
GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Chiral functionalization of 2,4,5,6-tetrachloro-1,3-dicyanobenzene by regioselective nucleophilic substitution of one or two chlorine atoms by optically pure (R)-(+)-1-naphthylethylamine (NEA), or by a glycine unit as

a spacer to (R)-NEA, enables the prepn. of brush-type chiral selectors, 2,5,6-trichloro-4-((R)-1'-naphthylethyl)amino-1,3-dicyanobenzene, 2,5-dichloro-4,6-bis-((R)-1'-naphthylethyl)amino-1,3-dicyanobenzene, 2,5,6-trichloro-4-N-[(R)-1'-naphthylethyl)aminomethylaminoacetyl]-1,3-dicyanobenzene, , and 2,5,6-trichloro-4-N-[(R)-1'-naphthylethyl)aminobutylaminoacetyl]-1,3-dicyanobenzene. By the introduction of the 3-aminopropyltriethoxysilyl (APTES) group, reactive intermediates, a regioisomeric pair of 2-(.gamma.-

triethoxysilylpropyl)amino-4-((R)-1'-naphthylethyl)amino-5,6-dichloro-1,3-dicyanobenzene and 6-(.gamma.-triethoxysilylpropyl)amino-4-((R)-1'-naphthylethyl)amino-2,5-dichloro-1,3-dicyanobenzene, 5-chloro-4,6-bis-((R)-1'-naphthylethyl)amino-2-(3'-triethoxysilylpropylamino)-1,3-dicyanobenzene, regioisomeric pair of 6-(.gamma.-

triethoxysilylpropyl)amino-4-N-[(R)-1'-naphthylethyl)aminomethylaminoacetyl]-2,5-dichloro-1,3-dicyanobenzene and 2-(.gamma.-

triethoxysilylpropyl)amino-4-N-[(R)-1'-naphthylethyl)aminomethylaminoacetyl]-5,6-dichloro-1,3-dicyanobenzene, and regioisomeric pair of 6-(.gamma.-triethoxysilylpropyl)amino-4-N-[(R)-1'-naphthylethyl)amino-n-butylaminoacetyl]-2,5-dichloro-1,3-dicyanobenzene and 2-(.gamma.-triethoxysilylpropyl)amino-4-N-[(R)-1'-naphthylethyl)amino-n-butylaminoacetyl]-5,6-dichloro-1,3-dicyanobenzene were obtained. Binding of these to silica gel afforded four novel chiral stationary phases

(CSPs)

I [R1 = Cl; R1= (R)-(+)-1-naphthylethylamino-] and II [R2 = CH3; R2 = (CH2)3CH3]. HPLC columns contg. CSPs with (R)-NEA directly linked to polysubstituted arom. ring I [R1 = Cl; R1= (R)-(+)-1-naphthylethylamino-] are not very effective in resolu. of most of the 23 racemic analytes, whereas the columns with distant .pi.-basic subunits II [R2 = CH3; R2 = (CH2)3CH3] exhibited higher **resolving** efficacy, in particular towards the iso-Pr esters of racemic N-3,5-dinitrobenzoyl-.alpha.-amino **acids**. Effective resolu. of test racemates reveals the importance of the presence of the hydrogen bond donor amido group and the distance between the persubstituted benzene ring in 2,4,5,6-tetrachloro-1,3-dicyanobenzene and the .pi.-basic naphthalene ring of (R)-NEA.

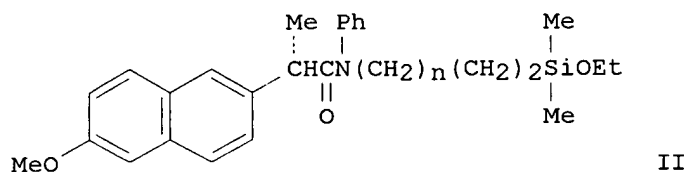
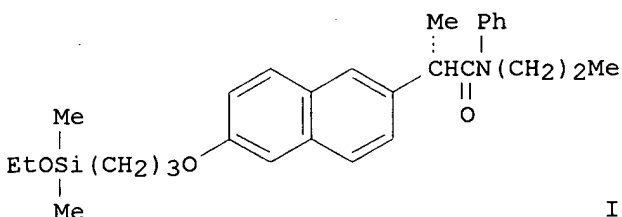
RE.CNT 27

RE

- (2) Allenmark, S; J Mater Sci 1997, V7, P1955 CAPLUS
 - (4) Cuntze, J; Helv Chim Acta 1997, V80, P897 CAPLUS
 - (5) Desiraju, G; Curr Opin Solid State Mater Sci 1997, V2, P451 CAPLUS
 - (6) Fornstedt, T; Chirality 1998, V10, P375 CAPLUS
 - (7) Gasparrini, F; J Chromatogr A 1996, V724, P79 CAPLUS
- ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 2 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1997:716854 CAPLUS
 DN 128:43149
 TI Preparation of a new HPLC chiral stationary phase from (S)-naproxen and application in elucidating chiral recognition models
 AU Hyun, Myung Ho; Kim, Kwang Ja; Jyung, Kyung Kyu
 CS Dep. Chemistry, Pusan National Univ., Pusan, 609-735, S. Korea
 SO Bull. Korean Chem. Soc. (1997), 18(10), 1085-1089
 CODEN: BKCSDE; ISSN: 0253-2964
 PB Korean Chemical Society
 DT Journal
 LA English
 GI



AB A new HPLC chiral stationary phase (silica gel-bound I) was prepd. from (S)-naproxen and compared with com. silica gel-bound chiral stationary phases II (n = 1, 9). The new stationary phase was applied to the resoln.

of a homologous series of N-(3,5-dinitrobenzoyl)-.alpha.-amino acid esters and a homologous series of N-(3,5-dinitrobenzoyl)-.alpha.-(4-alkylphenyl)alkylamines. The sepn. factors, .alpha., for **resolving** these homologous series remained const. throughout the range of the length of the alkyl substituent of the analytes, whereas those .alpha. factors from silica gel-bound II increased or decreased continuously. These results supported the chiral recognition models which

utilize the intercalation of the alkyl substituent of the racemic analytes

between the adjacent strands of silica gel-bound II to rationalize the increasing or decreasing trends of sepn. factors.

L7 ANSWER 3 OF 11 CAPLUS COPYRIGHT 2000 ACS
 AN 1996:750264 CAPLUS
 DN 126:152210
 TI Chiral recognition models for the liquid chromatographic resolution of .pi.-acidic racemates on a chiral stationary phase derived from N-phenyl-N-alkylamide of (S)-naproxen
 AU Hyun, Myung Ho; Na, Myeong Seon; Jin, Jong Sung
 CS Department Chemistry, Pusan National Univ., Pusan, 609-735, S. Korea
 SO J. Chromatogr., A (1996), 752(1+2), 77-84
 CODEN: JCRAEY; ISSN: 0021-9673
 PB Elsevier
 DT Journal
 LA English
 AB To elucidate the chiral recognition mechanism exerted by an

(S)-naproxen-derived chiral stationary phase (CSP) contg. a long tether consisting of a tertiary N-phenyl-N-undecyl amide linkage, a CSP with a short tether consisting of N-phenyl-N-Pr amide linkage was prepd. and homologous series of N-(3,5-dinitrobenzoyl)-.alpha.-amino alkyl esters

and

N-(3,5-dinitrobenzoyl)-.alpha.-(4-alkylphenyl)alkylamines were **resolved** on the two CSPs. Based on the chiral recognition trends for **resolving** homologous series of .pi.-**acidic** racemates on the two CSPs which differ only in the tether length and from the study of CPK space filling mol. models, chiral recognition models using the intercalation of the alkyl substituent of the analyte between the adjacent strands of bonded phase in rationalizing the dependence of the sepn. factors on the length of the alkyl substituent of the analyte and the tether length of the CSP were proposed.

L7 ANSWER 4 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1996:313305 CAPLUS

DN 125:96299

TI Improved chiral stationary phase derived from (S)-naproxen for the liquid chromatographic resolution of enantiomers

AU Ho Hyun, Myung; Seon Na, Myeong; Min, Chung-Sik

CS Department of Chemistry, Pusan National University, Pusan, 609-735, S. Korea

SO J. Chromatogr., A (1996), 732(2), 209-214

CODEN: JCRAEY; ISSN: 0021-9673

DT Journal

LA English

AB An (S)-naproxen-derived chiral stationary phase (CSP) contg. a tertiary N-Ph amide linkage was prepd. The CSP was applied to the resoln. of various .pi.-**acidic** racemates, including N-(3,5-dinitrobenzoyl) derivs. of .alpha.-amino esters and 3,5-dinitroanilide derivs. of anti-inflammatory drugs related to .alpha.-arylpropionic **acids**. From the comparison of the resoln. results this CSP showed greater enantioselectivities than any other (S)-naproxen-derived CSPs reported so far in **resolving** various .pi.-**acidic** racemates.

L7 ANSWER 5 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1995:742586 CAPLUS

DN 123:144624

TI Preparation of support-bound tartaric **acid**-amino **acid** monoamide derivative as reagent for chirality recognition and **resolving** agent for chromatography

IN Ooi, Takafumi; Kitahara, Hajime; Matsushita, Yasuhiro; Kisu, Naoko

PA Sumika Bunseki Center Kk, Japan

SO Jpn. Kokai Tokkyo Koho, 11 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 06298672	A2	19941025	JP 1993-89956	19930416

AB A reagent for chirality recognition has a structure comprising N-(3-carboxy-2,3-dihydroxypropionyl)amino **acid** N-(silylalkyl)amide (organosilane compd.) R1R2R3Si-R4-NHCO-R5-NR6COCH(OH)CH(OH)CO2H [R1, R2, R3 = alkyl, alkoxy, OH, halo, provided

that

at least one of R1 - R3 = alkoxy or halo; R4 = lower alkylene; R5 = residue derived by removing one NH2 and one CO2H group from optically active L- or D-amino **acid**; R6 = H, lower alkyl; or R5R6 = residue derived by removing one imino and one CO2H group from optically active L- or D-imino **acid**, provided that the remaining OH, NH, and NH2 in R5 may be substituted by Y-Z (wherein Y = CO or CONH and Z = lower alkyl, mono- or bicyclic aryl or aralkyl); each portion of tartaric **acid**, amino **acid**, and imino **acid** is in an

optically active form] grafted to an inorg. support having hydroxy groups on the surface. A chromatog. packing material comprises the compd. described above. This chiral recognition reagent is durable due to chem. stability, has in the structure both a ligand exchange part (which enables

direct sepn. of optical isomers such as amino **acids** and oxyacids) and a hydrogen-bonding interaction part (which enable sepn. of optical isomer derivs. such as amines, amino **acids**, and carboxylic **acids**), and are useful for optical resoln. of racemates. Thus, 200 g silica gel (av. grain diam. 5 .mu.m, av. pore diam. 120.ANG., and surface area 330 m2/g) was dried at 120.degree. in vacuo for 2 h and refluxed with 200 g 3-aminopropyltriethoxysilane in 1 L dry THF for 3 h to give 3-aminopropylated silica gel (0.94 mmol 3-aminopropyl group/1 g) which was condensed with Boc-Val-OH and after removing the Boc group, with 2,3-di-O-acetyl-L-tartaric **acid** followed by capping the residual amino group with Ac2O and hydrolysis

with

HClO4 in refluxing aq. MeCN to give

N-(N-3-carboxy-2,3-dihydroxypropionyl-

L-valyl)-3-aminopropylsilyl-grafted silica gel as a chromatog. packing material. A stainless steel column (inner diam. 4 mm .times. length 25 cm) packed with the latter packing material **resolved** racemic alcs. (binaphthol, uniconazole, and diniconazole), racemic amino **acid** deriv. [Ac-Val-OEt, DNB-Val-OMe (DNB = 3,5-dinitrobenzoyl), Ac-Phe-OMe, N-acetylphenylglycine, and N-DNB-phenylglycine], and racemic 1,1'-binaphthyl-2,2'-diyl hydrogen phosphate with sepn. coefficienty (.alpha. = K1'/K2', wherein K1', K2' = retention coefficienty for each enantiomer) of 1.037-1.158.

L7 ANSWER 6 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1995:653040 CAPLUS

DN 123:314413

TI Enantioselective Recognition by a New Chiral Stationary Phase at the Receptor Level

AU Gasparrini, Francesco; Misiti, Domenico; Villani, Claudio; Borchardt, Allen; Burger, Matthew T.; Still, W. Clark

CS Dipartimento di Studi di Chimica e Tecnologia delle Sostanze Biologicamente Attive, Universita La Sapienza, Rome, 00185, Italy

SO J. Org. Chem. (1995), 60(14), 4314-15

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA English

AB A new chiral stationary phase (CSP 1) for HPLC applications has been prepd. by covalent attachment of a synthetic C3-sym., O-allyl protected tyrosyl macrocycle to .gamma.-mercaptopropyl silica gel. CSP 1 shows exceptionally high levels of enantioselectivity for Boc-protected amino **acid** derivs., with sepn. factors in the 9-43 range using org. eluents; multiple H-bond interactions between the cup-shaped, immobilized C3-macrocycle and enantiomeric guests are involved in the recognition process. The preferential retention of L-Boc-protected amino **acids** is reversed for .pi.-**acidic** 3,5-dinitrobenzoylated amino **acid** derivs., suggesting addnl. binding modes based on .pi.-stacking interactions. Chromatog. data collected under reversed-phase conditions show that the macrocyclic receptor is capable

of

enantioselective recognition also in aq. media. In addn. to enantiomeric and diastereomeric resolns. of simple peptidic substrates, CSP 1 can **resolve** a large variety of racemic compds. having different types and combinations of functionalities.

L7 ANSWER 7 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1995:28519 CAPLUS

DN 122:122212

TI Comparison between silica-bonded chiral stationary phases derived from 3,5-disubstituted N-benzoyl-(S)-phenylalanine and (S)-cyclohexylalanine

in

the resolution of racemic compounds by liquid chromatography

AU Oliveros, Laureano; Minguillon, Cristina; Gonzalez, Teresa

CS Conservatoire National des Arts et Metiers, Laboratoire de Chimie

Generale, 292 Rue Saint-Martin, Paris, 75141/03, Fr.

SO J. Chromatogr., A (1994), 672(1-2), 59-65

CODEN: JCRAEY

DT Journal

LA English

AB A study was made of the role of the phenylalanine Ph ring in the enantioselectivity of several chiral stationary phases (CSPs) whose

chiral selectors consist of several N-(3,5-disubstituted)benzoyl derivs. of this amino **acid** covalently bonded to silica gel. Racemic compds. with .pi.-acceptor, .pi.-donor or both characters were **resolved** on two series of CSPs derived from N-(3,5-dimethyl)benzoyl, N-(3,5-dimethoxy)benzoyl and N-(3,5-dinitro)benzoyl-(S)-phenylalanine and (S)-cyclohexylalanine. In all instances the best enantioselectivities were obtained with CSPs derived from (S)-cyclohexylalanine. The Ph ring in the phenylalanine moiety does not have an electronic role in the recognition of the racemic compd. by the chiral selector on the CSP, a nonclassical .pi.-.pi. interaction between the 3,5-dinitrobenzoyl group

in the racemic compd. and in the CSP acts in the resolu. of N-(3,5-dinitro)benzoyl derivs. of amino **acids** on CSPs with the same group and the change in the arrangement of solutes in the diastereomeric solute-stationary phase complexes can take place without

an inversion of the elution order of enantiomers.

L7 ANSWER 8 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1995:14309 CAPLUS

DN 122:95579

TI Diphenylethanediamine (DPEDA) derivatives as chiral selectors: IV. A comparison of 3,5-dinitrobenzoylated (S,S)- and (S,R)-DPEDA-derived

chiral stationary phases with Pirkle's standard (R)-phenylglycine-derived phase in normal phase HPLC

AU Maier, Norbert M.; Uray, Georg; Kleidernigg, Oliver P.; Lindner, Wolfgang

CS Inst. Org. Chem., Karl Franzens Univ., Graz, Austria

SO Chirality (1994), 6(2), 116-28

CODEN: CHRLEP; ISSN: 0899-0042

DT Journal

LA English

AB Undecanoyl bound 3,5-dinitrobenzoyl-(S,R)-1,2-diphenylethane-1,2-diamine [(1S,2R)-DNB-DPEDA] as chiral selector (SO) was synthesized and used as a chiral stationary phase (CSP II) for normal-phase enantioselective HPLC. It is compared with the already published diastereomeric (1S,2S)-DNB-DPEDA-derived CSP I and with the std. Pirkle DNB-(R)-phenylglycine-derived CSP III. Chromatog. data for .apprx.100 racemic analytes reveal that CSP II is able to sep. esp. well enantiomers of derivatized arom. carboxylic **acids** and analytes having a benzyl substituent bound at the chiral center. However, CSP I is

superior to CSP II and III in its general applicability and its ability to **resolve** enantiomers of heterocyclic drugs.

L7 ANSWER 9 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1994:629986 CAPLUS

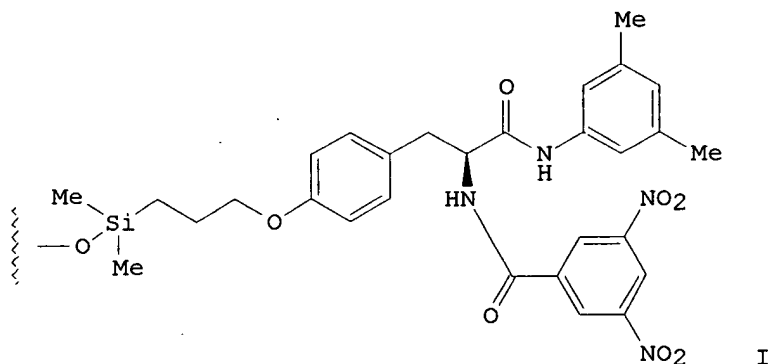
DN 121:229986

TI A new chiral stationary phase bearing both .pi.-acidic and .pi.-basic sites derived from (S)-tyrosine for the liquid chromatographic resolution of racemates

AU Hyun, Myung Ho; Min, Chung Sik

CS Dep. Chem., Pusan Natl. Univ., Pusan, 609-735, S. Korea

SO Chem. Lett. (1994), (8), 1463-6



AB A new chiral stationary phase I, bearing both .pi.-**acidic** and .pi.-basic sites, has been prepd. from (S)-tyrosine and used in chiral liq. chromatog. I is useful in **resolving** a variety of either .pi.-basic or .pi.-**acidic** racemates.

L7 ANSWER 10 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1987:176817 CAPLUS

DN 106:176817

TI Enantiomer separation by HPLC with some urea derivatives of L-valine as novel chiral stationary phases

AU Oi, Naobumi; Kitahara, Hajimu

CS Sumika Chem. Anal. Serv., Ltd., Osaka, 554, Japan

SO J. Liq. Chromatogr. (1986), 9(2-3), 511-17

CODEN: JLCHD8; ISSN: 0148-3919

DT Journal

LA English

AB Two novel chiral stationary phases derived from N-(S)-1-(.alpha.-naphthyl)ethylaminocarbonyl-L-valine and the corresponding (R)-epimer chem. bonded to .gamma.-aminopropyl silanized silica, which contain two asym. carbon atoms attached to two nitrogen atoms of the urea group, were prepd. These phases showed excellent enantioselectivity for derivs. of amino **acid**, amine, carboxylic **acid** and alc. enantiomers. Some alc. and ester enantiomers were **resolved** directly without any prederivatization upon these phases.

L7 ANSWER 11 OF 11 CAPLUS COPYRIGHT 2000 ACS

AN 1984:491414 CAPLUS

DN 101:91414

TI A chiral stationary phase for the facile resolution of amino acids, amino alcohols and amines as the N-3,5-dinitrobenzoyl derivatives

AU Pirkle, William H.; Hyun, Myung Ho

CS Sch. Chem. Sci., Univ. Illinois, Urbana, IL, 61801, USA

SO J. Org. Chem. (1984), 49(17), 3043-6

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA English

AB A chiral stationary phase derived from .alpha.-(6,7-dimethyl-1-naphthyl)isobutylamine is quite effective for the liq. chromatog. sepn.

of

the enantiomers of the N-3,5-dinitrobenzoyl derivs. of .alpha.-amino **acids**, their esters and amides, amino alcs., and amines. For example, the chromatog. sepn. factor for the enantiomers of derivatized

Me

phenylalanate is 4.73, the band shapes are excellent, and the resolu. value is 16.5. Addnl., the 3,5-dinitrobenzoates of a no. of secondary alcs. are **resolvable** on this chiral phase. An analogous stationary phase derived from .alpha.-(1-naphthyl)ethylamine performs less well for the amino **acid** ester derivs. but is superior for a few of the amine derivs.

AN 1995:742586 CAPLUS
 DN 123:144624
 TI Preparation of support-bound **tartaric acid**-amino
acid monoamide derivative as reagent for chirality recognition and
resolving agent for chromatography
 IN Ooi, Takafumi; Kitahara, Hajime; Matsushita, Yasuhiro; Kisu, Naoko
 PA Sumika Bunseki Center Kk, Japan
 SO Jpn. Kokai Tokkyo Koho, 11 pp.
 CODEN: JKXXAF
 DT Patent
 LA Japanese
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 06298672	A2	19941025	JP 1993-89956	19930416

AB A reagent for chirality recognition has a structure comprising
 N-(3-carboxy-2,3-dihydroxypropionyl)amino **acid**
 N-(silylalkyl)amide (organosilane compd.) R1R2R3Si-R4-NHCO-R5-
 NR6COCH(OH)CH(OH)CO2H [R1, R2, R3 = alkyl, alkoxy, OH, halo, provided
 that
 at least one of R1 - R3 = alkoxy or halo; R4 = lower alkylene; R5 =
 residue derived by removing one NH2 and one CO2H group from optically
 active L- or D-amino **acid**; R6 = H, lower alkyl; or R5R6 =
 residue derived by removing one imino and one CO2H group from optically
 active L- or D-imino **acid**, provided that the remaining OH, NH,
 and NH2 in R5 may be substituted by Y-Z (wherein Y = CO or CONH and Z =
 lower alkyl, mono- or bicyclic aryl or aralkyl); each portion of
tartaric acid, amino **acid**, and imino
acid is in an optically active form] grafted to an inorg. support
 having hydroxy groups on the surface. A chromatog. packing material
 comprises the compd. described above. This chiral recognition reagent is
 durable due to chem. stability, has in the structure both a ligand
 exchange part (which enables direct sepn. of optical isomers such as
 amino
acids and oxyacids) and a hydrogen-bonding interaction part (which
 enable sepn. of optical isomer derivs. such as amines, amino **acids**
 , and carboxylic **acids**), and are useful for optical resoln. of
 racemates. Thus, 200 g silica gel (av. grain diam. 5 .mu.m, av. pore
 diam. 120.ANG., and surface area 330 m2/g) was dried at 120.degree. in
 vacuo for 2 h and refluxed with 200 g 3-aminopropyltriethoxysilane in 1 L
 dry THF for 3 h to give 3-aminopropylated silica gel (0.94 mmol
 3-aminopropyl group/1 g) which was condensed with Boc-Val-OH and after
 removing the Boc group, with 2,3-di-O-acetyl-L-**tartaric**
acid followed by capping the residual amino group with Ac2O and
 hydrolysis with HClO4 in refluxing aq. MeCN to give N-(N-3-carboxy-2,3-
 dihydroxypropionyl-L-valyl)-3-aminopropylsilyl-grafted silica gel as a
 chromatog. packing material. A stainless steel column (inner diam. 4 mm
 .times. length 25 cm) packed with the latter packing material
resolved racemic alcs. (binaphthol, uniconazole, and
 diniconazole), racemic amino **acid** deriv. [Ac-Val-OEt,
 DNB-Val-OMe (DNB = 3,5-dinitrobenzoyl), Ac-Phe-OMe,
 N-acetylphenylglycine,
 and N-DNB-phenylglycine], and racemic 1,1'-binaphthyl-2,2'-diyl hydrogen
 phosphate with sepn. coefficient (.alpha. = K1'/K2', wherein K1', K2' =
 retention coefficient for each enantiomer) of 1.037-1.158.

=> d hitstr

L12 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2000 ACS

IT **69632-32-2P**

RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)

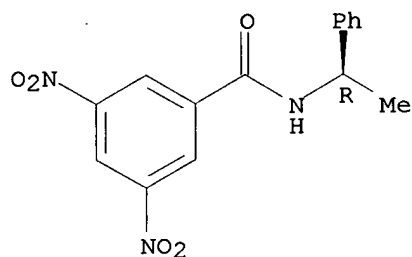
(prepn. by chromatog. optical resoln. with support-bound **tartaric acid-amino acid** monoamide derivs.

as reagents for chirality recognition and chromatog. **resolving** agents)

RN 69632-32-2 CAPLUS

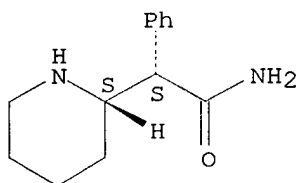
CN Benzamide, 3,5-dinitro-N-[(1R)-1-phenylethyl]- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



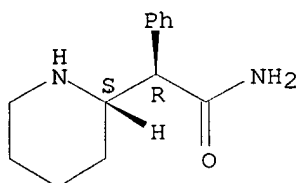
AN 1995:92409 CAPLUS
 DN 122:122242
 TI Determination of enantiomeric composition of 2-phenyl-2-(2-piperidyl)acetamide. A routine method for evaluation of enantiomeric purity of primary amides
 AU Jursic, Branko S.; Zdravkovski, Zoran; Zuanic, Miljenko
 CS Dep. Chem., Univ. New Orleans, New Orleans, LA, 70148, USA
 SO Tetrahedron: Asymmetry (1994), 5(9), 1711-16
 CODEN: TASYE3; ISSN: 0957-4166
 DT Journal
 LA English
 AB Several NMR chiral resolving agents have successfully been used to demonstrate their usefulness for the detn. of the enantiomeric compn. of the title compd.
 IT **160707-38-0 160707-39-1**
 RL: ANT (Analyte); PRP (Properties); ANST (Analytical study)
 (detn. of, in enantiomer mixts. by NMR spectroscopy)
 RN 160707-38-0 CAPLUS
 CN 2-Piperidineacetamide, .alpha.-phenyl-, [S-(R*,R*)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



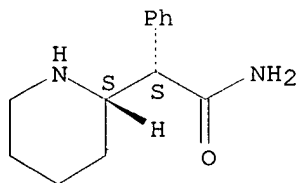
RN 160707-39-1 CAPLUS
 CN 2-Piperidineacetamide, .alpha.-phenyl-, [S-(R*,S*)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



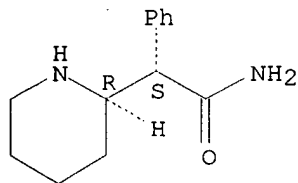
AN 1973:526273 CAPLUS
 DN 79:126273
 TI Syntheses of thioacetamide derivatives. I
 AU Sanno, Yasushi; Kanai, Yoshio; Nohara, Akira; Hirata, Takeo
 CS Takeda Chem. Ind., Ltd., Osaka, Japan
 SO Takeda Kenkyusho Ho (1973), 32(2), 125-9
 CODEN: TAKHAA
 DT Journal
 LA Japanese
 AB In order to find a new nonanticholinergic antiulcer agent, 2-phenyl-2-[2-(N-methylpyridinium)[thioacetamide iodide, 2-phenyl-2-(2-piperidyl)thioacetamide, 2-phenyl-2-(3-pyridyl)thioacetamide, 1-methyl-4-(2-pyridyl)piperidine-4-thioformamide, DL-phenylalaninethioamide, 2-[2-(1-benzylimidazolyl)]thioacetamide, and 3-(2-pyridyl)thiopropionamide were prepd.
 IT **50288-62-5P 50288-63-6P**
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (prepn. of)
 RN 50288-62-5 CAPLUS
 CN 2-Piperidineacetamide, .alpha.-phenyl-, (R*,R*)- (9CI) (CA INDEX NAME)

Relative stereochemistry.



RN 50288-63-6 CAPLUS
 CN 2-Piperidineacetamide, .alpha.-phenyl-, (R*,S*)- (9CI) (CA INDEX NAME)

Relative stereochemistry.



=> s c13h18n2o/mf

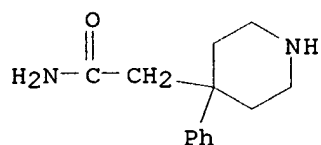
L1 1461 C13H18N2O/MF

=> s l1 and piperidineacetamide

L2 2955 PIPERIDINEACETAMIDE
13 L1 AND PIPERIDINEACETAMIDE

=> d 1-13

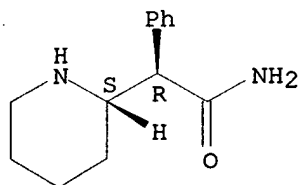
L2 ANSWER 1 OF 13 REGISTRY COPYRIGHT 1998 ACS
RN 200272-07-7 REGISTRY
CN 4-Piperidineacetamide, 4-phenyl- (9CI) (CA INDEX NAME)
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS, USPATFULL



1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 2 OF 13 REGISTRY COPYRIGHT 1998 ACS
RN 160707-39-1 REGISTRY
CN 2-Piperidineacetamide, .alpha.-phenyl-, [S-(R*,S*)]- (9CI)
(CA INDEX NAME)
FS STEREOSEARCH
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS

Absolute stereochemistry.

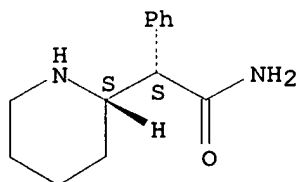


1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 3 OF 13 REGISTRY COPYRIGHT 1998 ACS
RN 160707-38-0 REGISTRY
CN 2-Piperidineacetamide, .alpha.-phenyl-, [S-(R*,R*)]- (9CI)

(CA INDEX NAME)
FS STEREOSEARCH
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS

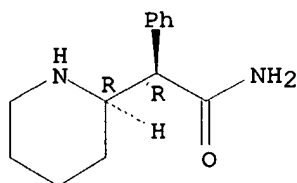
Absolute stereochemistry.



1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 4 OF 13 REGISTRY COPYRIGHT 1998 ACS
RN 160707-37-9 REGISTRY
CN 2-Piperidineacetamide, .alpha.-phenyl-, [R-(R*,R*)]- (9CI)
(CA INDEX NAME)
FS STEREOSEARCH
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS

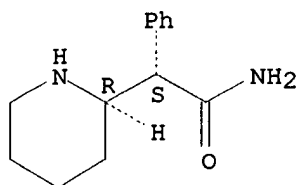
Absolute stereochemistry.



1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

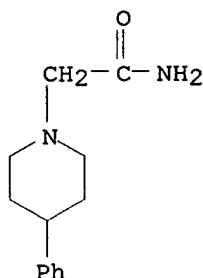
L2 ANSWER 5 OF 13 REGISTRY COPYRIGHT 1998 ACS
RN 160707-36-8 REGISTRY
CN 2-Piperidineacetamide, .alpha.-phenyl-, [R-(R*,S*)]- (9CI)
(CA INDEX NAME)
FS STEREOSEARCH
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS

Absolute stereochemistry.



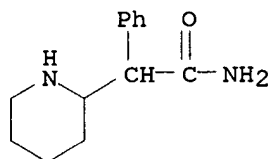
1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 6 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 84863-82-1 REGISTRY
 CN **1-Piperidineacetamide, 4-phenyl- (9CI)** (CA INDEX NAME)
 FS 3D CONCORD
 MF **C13 H18 N2 O**
 LC STN Files: BEILSTEIN*, CA, CAPLUS, CJACS, TOXLIT
 (*File contains numerically searchable property data)



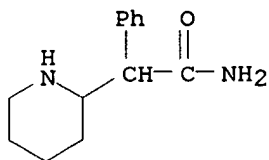
1 REFERENCES IN FILE CA (1967 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 7 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 83024-87-7 REGISTRY
 CN **2-Piperidineacetamide, .alpha.-phenyl-, (R*,R*)-, labeled with deuterium (9CI)** (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN **2-Piperidineacetamide, .alpha.-phenyl-, (R*,R*)-(.+-.)-, labeled with deuterium**
 MF **C13 H18 N2 O**
 CI COM
 LC STN Files: CA, CAPLUS
 IL XH-2



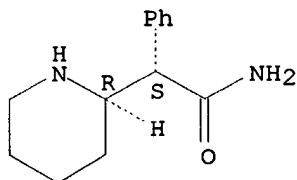
1 REFERENCES IN FILE CA (1967 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 8 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 82993-79-1 REGISTRY
 CN **2-Piperidineacetamide, .alpha.-phenyl-, (R*,S*)-, labeled with deuterium (9CI)** (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN **2-Piperidineacetamide, .alpha.-phenyl-, (R*,S*)-(.+-.)-, labeled with deuterium**
 MF **C13 H18 N2 O**
 CI COM
 IL XH-2



L2 ANSWER 9 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 50288-63-6 REGISTRY
 CN **2-Piperidineacetamide, .alpha.-phenyl-, (R*,S*)-** (9CI)
 (CA INDEX NAME)
 FS STEREOSEARCH
 MF **C13 H18 N2 O**
 LC STN Files: BEILSTEIN*, CA, CAPLUS
 (*File contains numerically searchable property data)

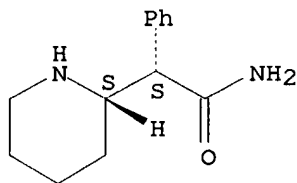
Relative stereochemistry.



1 REFERENCES IN FILE CA (1967 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

L2 ANSWER 10 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 50288-62-5 REGISTRY
 CN **2-Piperidineacetamide, .alpha.-phenyl-, (R*,R*)-** (9CI)
 (CA INDEX NAME)
 FS STEREOSEARCH
 MF **C13 H18 N2 O**
 LC STN Files: BEILSTEIN*, CA, CAPLUS
 (*File contains numerically searchable property data)

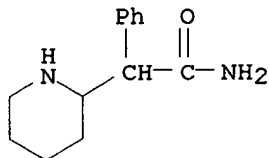
Relative stereochemistry.



1 REFERENCES IN FILE CA (1967 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

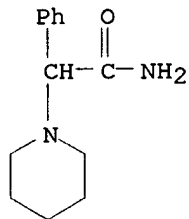
L2 ANSWER 11 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 19395-39-2 REGISTRY
 CN **2-Piperidineacetamide, .alpha.-phenyl-** (6CI, 7CI, 8CI, 9CI)
 (CA INDEX NAME)
 OTHER NAMES:
 CN 2-Phenyl-2-(2-piperidyl)acetamide
 FS 3D CONCORD
 MF **C13 H18 N2 O**
 CI COM
 LC STN Files: BEILSTEIN*, CA, CAOLD, CAPLUS, CHEMLIST

(*File contains numerically searchable property data)
 Other Sources: EINECS**
 (**Enter CHEMLIST File for up-to-date regulatory information)



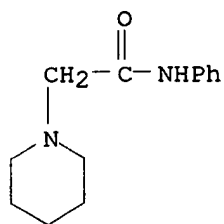
2 REFERENCES IN FILE CA (1967 TO DATE)
 2 REFERENCES IN FILE CAPLUS (1967 TO DATE)
 4 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

L2 ANSWER 12 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 7253-67-0 REGISTRY
 CN **1-Piperidineacetamide, .alpha.-phenyl-** (6CI, 8CI, 9CI)
 (CA INDEX NAME)
 FS 3D CONCORD
 MF **C13 H18 N2 O**
 CI COM
 LC STN Files: BEILSTEIN*, CA, CAOLD, CAPLUS, CASREACT, CHEMCATS,
 CHEMLIST, SPECINFO
 (*File contains numerically searchable property data)
 Other Sources: EINECS**
 (**Enter CHEMLIST File for up-to-date regulatory information)



2 REFERENCES IN FILE CA (1967 TO DATE)
 2 REFERENCES IN FILE CAPLUS (1967 TO DATE)
 3 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

L2 ANSWER 13 OF 13 REGISTRY COPYRIGHT 1998 ACS
 RN 4671-97-0 REGISTRY
 CN **1-Piperidineacetamide, N-phenyl-** (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN 1-Piperidineacetanilide (6CI, 7CI, 8CI)
 OTHER NAMES:
 CN 2-Piperidinoacetanilide
 CN DG 1
 CN Piperidinoacetanilide
 FS 3D CONCORD
 MF **C13 H18 N2 O**
 CI COM
 LC STN Files: BEILSTEIN*, BIOSIS, CA, CAOLD, CAPLUS, RTECS*, TOXLIT
 (*File contains numerically searchable property data)



20 REFERENCES IN FILE CA (1967 TO DATE)
7 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
20 REFERENCES IN FILE CAPLUS (1967 TO DATE)
6 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

=> fil ca

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	25.04	25.19

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FILE COVERS 1967 - 14 Apr 1998 (980414/ED) VOL 128 ISS 16

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 160707-39-1 or 160707-38-0 or 160707-37-9 or 160707-36-8 or 83024-87-7
or 82993-79-1 or 50288-63-6 or 80288-62-5 or 19395-39-2

1 160707-39-1
1 160707-38-0
1 160707-37-9
1 160707-36-8
1 83024-87-7
0 82993-79-1
1 50288-63-6
0 80288-62-5
2 19395-39-2
L3 4 160707-39-1 OR 160707-38-0 OR 160707-37-9 OR 160707-36-8
OR 83024-87-7 OR 82993-79-1 OR 50288-63-6 OR 80288-62-5
OR 19395-39-2

=> d bib abs 1-4

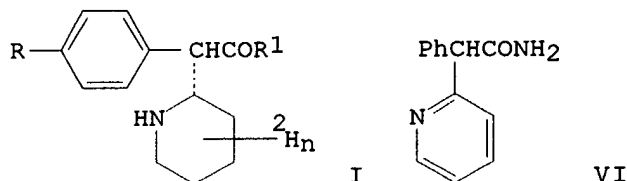
L3 ANSWER 1 OF 4 CA COPYRIGHT 1998 ACS
AN 122:122242 CA
TI Determination of enantiomeric composition of 2-phenyl-2-(2-piperidyl)acetamide. A routine method for evaluation of enantiomeric purity of primary amides
AU Jursic, Branko S.; Zdravkovski, Zoran; Zuanic, Miljenko
CS Dep. Chem., Univ. New Orleans, New Orleans, LA, 70148, USA
SO Tetrahedron: Asymmetry (1994), 5(9), 1711-16
CODEN: TASYE3; ISSN: 0957-4166
DT Journal
LA English
AB Several NMR chiral resolving agents have successfully been used to demonstrate their usefulness for the detn. of the enantiomeric compn. of the title compd.

L3 ANSWER 2 OF 4 CA COPYRIGHT 1998 ACS

AD 241.74

AN 97:144733 CA
 TI Synthesis of deuterium-labeled methylphenidate, p-hydroxymethylphenidate, ritalinic acid, and p-hydroxyritalinic acid
 AU Patrick, Kennerly; Kilts, Clinton; Breese, George
 CS Biol. Sci. Res. Cent., Univ. North Carolina, Chapel Hill, NC, 27514, USA
 SO J. Labelled Compd. Radiopharm. (1982), 19(4), 485-90
 CODEN: JLCRD4; ISSN: 0362-4803
 DT Journal
 LA English
 GI

6D 466, J6



AB In the preps. of the title compds. (I; R = H, OH, R1 = OMe; R = H, OH, R1 = OH) (II-V, resp.), all possible combinations of 2H on the piperidine ring were obtained, the most abundant being the pentadeuterated product. Deuteration of the amide VI gave a 70:30 erythro-threo mixt. I (R = H, R1 = NH2) which after KOH epimerization and treatment with HCl gave 74% IV.HCl. Subsequent esterification of IV.HCl gave 89% II.HCl. III.HCl and V.HBr were prepd. by modification of a previous method (1981). II-V were prepd. as internal stds. for mass fragmentog. assays of methylphenidate and its metabolites.

L3 ANSWER 3 OF 4 CA COPYRIGHT 1998 ACS

AN 79:126273 CA

TI Syntheses of thioacetamide derivatives. I

AU Sanno, Yasushi; Kanai, Yoshio; Nohara, Akira; Hirata, Takeo

CS Takeda Chem. Ind., Ltd., Osaka, Japan

SO Takeda Kenkyusho Ho (1973), 32(2), 125-9

CODEN: TAKHAA

DT Journal

LA Japanese

AB In order to find a new nonanticholinergic antiulcer agent, 2-phenyl-2-[2-(N-methylpyridinium)[thioacetamide iodide, 2-phenyl-2-(2-piperidyl)thioacetamide, 2-phenyl-2-(3-pyridyl)thioacetamide, 1-methyl-4-(2-pyridyl)piperidine-4-thioformamide, DL-phenylalaninethioamide, 2-[2-(1-benzylimidazolyl)]thioacetamide, and 3-(2-pyridyl)thiopropionamide were prepd.

L3 ANSWER 4 OF 4 CA COPYRIGHT 1998 ACS

AN 69:77026 CA

TI Condensed 2-azetidinones. II. Isomeric 3-phenyl-1-azabicyclo[4,2,0]octan-2-ones

AU Moll, F.

CS Univ. Tuebingen, Tuebingen, Ger.

SO Arch. Pharm. (Weinheim) (1968), 301(4), 250-62

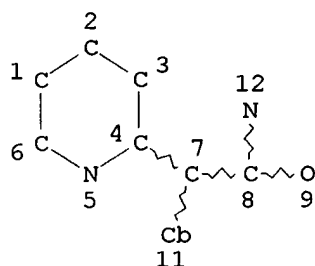
CODEN: APBDAJ

DT Journal

LA German

AB .alpha.-Phenyl-.alpha.-pyrid-2-ylacetonitrile was converted into the corresponding amide, and reduced to .alpha.-phenyl-.alpha.-piperid-2-

ylacetamide, which was treated with 18% HCl 6 hrs. at 110.degree. to give .alpha.-phenyl-.alpha.-piperid-2-ylacetic acid-HCl (I). Attempts to remove the HCl from I gave mainly piperidylacetic acid. .alpha.-Ethyl-.alpha.-phenyl-.alpha.-piperid-2-ylacetamide was similarly obtained, but could not be converted into .alpha.-ethyl-.alpha.-phenyl-.alpha.-piperid-2-ylacetic acid-HCl. .alpha.-Phenyl-.alpha.-piperid-2-ylacetyl chloride-HCl (II) was prepd. from .alpha.-phenyl-.alpha.-piperid-2-ylacetic acid and SOCl₂. II was treated with Et₃N 24 hrs. at room temp. to give 3-phenyl-1-azabicyclo[4.2.0]octan-2-one (III), also obtained by treating I with dicyclohexylcarbodiimide. The isomers of II were sepd. by gas chromatog. on polyethylene glycol (20,000) at 220.degree., 106 ml./min. He. The cis isomer, m. 70.degree., had a retention time of 20 min. and the trans isomer 23 min. III was also prepd. by photolysis of phenylglyoxylpiperidine dianzo ketone as described by E. J. Corey and A. M. Felix (1965). The trans isomer was the major product in this reaction.



ENTER (DIS), GRA, NOD, BON OR ?:end.

L1 STRUCTURE CREATED

=> s 11

SAMPLE SEARCH INITIATED 10:49:28

SAMPLE SCREEN SEARCH COMPLETED - 2852 TO ITERATE

35.1% PROCESSED 1000 ITERATIONS

0 ANSWERS

INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)

SEARCH TIME: 00.00.02

FULL FILE PROJECTIONS: ONLINE **COMPLETE**

BATCH **COMPLETE**

PROJECTED ITERATIONS: 53841 TO 60239

PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1

=> s 11 ful

FULL SEARCH INITIATED 10:49:40

FULL SCREEN SEARCH COMPLETED - 54785 TO ITERATE

59.7% PROCESSED 32715 ITERATIONS

13 ANSWERS

90.5% PROCESSED 49605 ITERATIONS

20 ANSWERS

100.0% PROCESSED 54785 ITERATIONS

32 ANSWERS

SEARCH TIME: 00.00.58

L3 32 SEA SSS FUL L1

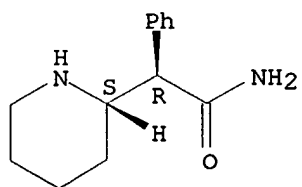
=> d scan

L3 32 ANSWERS REGISTRY COPYRIGHT 1998 ACS

IN 2-Piperidineacetamide, .alpha.-phenyl-, [S-(R*,S*)]- (9CI)

MF C13 H18 N2 O

Absolute stereochemistry.



=> fil ca

COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	115.42	115.57

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FILE COVERS 1967 - 24 Mar 1998 (980324/ED) VOL 128 ISS 13

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 13

L4 12 L3

=> s 14 and threo?

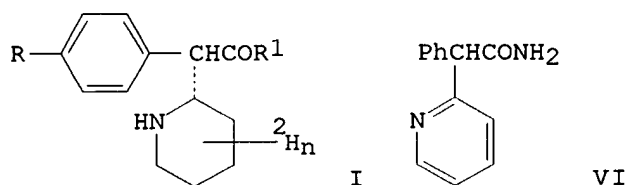
35471 THREO?
 L5 3 L4 AND THREO?

=> d bib abs 1-3

L5 ANSWER 1 OF 3 CA COPYRIGHT 1998 ACS
 AN 124:219394 CA
 TI Synthesis and pharmacology of potential cocaine antagonists. 2.
 Structure-activity relationship studies of aromatic ring-substituted
 methylphenidate analogs
 AU Deutsch, Howard M.; Shi, Qing; Gruszecka-Kowalik, Ewa; Schweri,
 Margaret M.
 CS School of Chemistry and Biochemistry, Georgia Institute of
 Technology, Atlanta, GA, 30332-0400, USA
 SO J. Med. Chem. (1996), 39(6), 1201-9
 CODEN: JMCMAR; ISSN: 0022-2623
 DT Journal
 LA English
 OS CASREACT 124:219394; CJACS-IMAGE; CJACS
 AB As part of a program to develop medications which can block the
 binding of cocaine to the dopamine transporter, yet spare dopamine
 uptake, a series of arom. ring-substituted methylphenidate derivs.
 was synthesized and tested for inhibitory potency in [3H]WIN 35,428
 binding and [3H]dopamine uptake assays using rat striatal tissue.
 Synthesis was accomplished by alkylation of 2-bromopyridine with
 anions derived from various substituted phenylacetone nitriles. In
 most cases, erythro compds. were markedly less potent than the
 corresponding (.-.)-threo-methylphenidate (TMP; Ritalin)

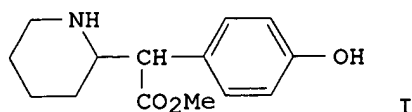
derivs. The ortho-substituted compds. were much less potent than the corresponding meta- and/or para-substituted derivs. The most potent compd. against [3H]WIN 35,428 binding, m-bromo-TMP, was 20-fold more potent than the parent compd., whereas the most potent compd. against [3H]dopamine uptake, m,p-dichloro-TMP, was 32-fold more potent. **Threo** derivs. with m- or p-halo substituents were more potent than TMP, while electron-donating substituents caused little change or a small loss of potency. All of the derivs. had Hill coeffs. approaching unity, except m,p-dichloro-TMP, which had an nH of 2.0. Although the potency of the (.+.-)-methylphenidate derivs. in the two assays was highly correlated ($R^2 = 0.986$), the compds. m-chloro-, m-methyl-, and p-iodo-TMP were 4-5-fold more potent at inhibiting [3H]WIN 35,428 binding than [3H]dopamine uptake (cocaine has a ratio of 2.3). These and other compds. may be promising candidates for further testing as potential partial agonists or antagonists of cocaine.

L5 ANSWER 2 OF 3 CA COPYRIGHT 1998 ACS
 AN 97:144733 CA
 TI Synthesis of deuterium-labeled methylphenidate, p-hydroxymethylphenidate, ritalinic acid, and p-hydroxyritalinic acid
 AU Patrick, Kennerly; Kilts, Clinton; Breese, George
 CS Biol. Sci. Res. Cent., Univ. North Carolina, Chapel Hill, NC, 27514, USA
 SO J. Labelled Compd. Radiopharm. (1982), 19(4), 485-90
 CODEN: JLCRD4; ISSN: 0362-4803
 DT Journal
 LA English
 GI



AB In the preps. of the title compds. (I; R = H, OH, R1 = OMe; R = H, OH, R1 = OH) (II-V, resp.), all possible combinations of 2H on the piperidine ring were obtained, the most abundant being the pentadeuterated product. Deuteration of the amide VI gave a 70:30 erythro-**threo** mixt. I (R = H, R1 = NH2) which after KOH epimerization and treatment with HCl gave 74% IV.HCl. Subsequent esterification of IV.HCl gave 89% II.HCl. III.HCl and V.HBr were prepd. by modification of a previous method (1981). II-V were prepd. as internal stds. for mass fragmentog. assays of methylphenidate and its metabolites.

L5 ANSWER 3 OF 3 CA COPYRIGHT 1998 ACS
 AN 95:125869 CA
 TI Synthesis and pharmacology of hydroxylated metabolites of methylphenidate
 AU Patrick, Kennerly S.; Kilts, Clinton D.; Breese, George R.
 CS Sch. Med., Univ. North Carolina, Chapel Hill, NC, 27514, USA
 SO J. Med. Chem. (1981), 24(10), 1237-40
 CODEN: JMCMAR; ISSN: 0022-2623
 DT Journal
 LA English
 GI



AB Me **threo**-dl- (I) [78708-74-4] and erythro-dl-p-hydroxymethylphenidate-HCl (II) [78708-67-5] and their resp. deesterified products III [78708-75-5] and IV [78708-76-6] were synthesized and tested for dopaminergic activity in rats. The locomotor response to I was greater than that to II, ritalin (V) [298-59-9], or erythro-dl-methylphenidate-HCl [23644-60-2], suggesting that I may play a role in the pharmacol. of V in the hyperkinetic syndrome in children. **threo**-dl-Ritalinic acid-HBr [78708-68-6], erythro-dl-ritalinic acid-HBr [78779-59-6], III, and IV produced small increases in locomotor activity relative to their Me esters and the responses were not appreciably affected by stereochem. or para-hydroxylation.

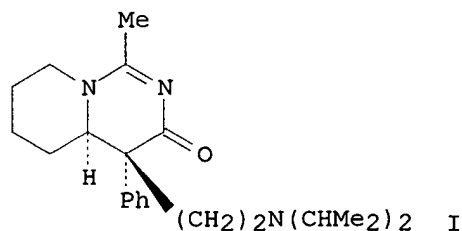
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L6 9 L4 NOT L5

=> d bib abs 1-9

L6 ANSWER 1 OF 9 CA COPYRIGHT 1998 ACS
 AN 122:122242 CA
 TI Determination of enantiomeric composition of 2-phenyl-2-(2-piperidyl)acetamide. A routine method for evaluation of enantiomeric purity of primary amides
 AU Jursic, Branko S.; Zdravkovski, Zoran; Zuanic, Miljenko
 CS Dep. Chem., Univ. New Orleans, New Orleans, LA, 70148, USA
 SO Tetrahedron: Asymmetry (1994), 5(9), 1711-16
 CODEN: TASYE3; ISSN: 0957-4166
 DT Journal
 LA English
 AB Several NMR chiral resolving agents have successfully been used to demonstrate their usefulness for the detn. of the enantiomeric compn. of the title compd.

L6 ANSWER 2 OF 9 CA COPYRIGHT 1998 ACS
 AN 108:156546 CA
 TI HPLC method development for a new antiarrhythmic drug
 AU Roston, Daryl A.
 CS Searle Res. Dev., Skokie, IL, 60077, USA
 SO J. Liq. Chromatogr. (1987), 10(15), 3427-40
 CODEN: JLCHD8; ISSN: 0148-3919
 DT Journal
 LA English
 GI



AB A HPLC method for the detn. of a new antiarrhythmic drug (I) was developed. Selectivity optimization for I and several synthetic process intermediates with reversed-phase HPLC conditions is described. Also, the use of electrochem. and UV absorption detection for I samples was evaluated. A Supelco LC-18-DB column and a mobile phase, prepd. by adding 20 mL Et₃N to 1800 mL water and adjusting the pH with concd. H₃PO₄, and mixing with MeCN (84:16), were used. The developed method was validated for generation of assay data for chem. lots of I.

L6 ANSWER 3 OF 9 CA COPYRIGHT 1998 ACS

AN 107:176063 CA

TI Preparation of octahydropyrido [1,2-c]pyrimidinones and hexahydropyrido[1,2-c]pyrimidinediones as antiarrhythmics

IN Fowler, Kerry W.; Chorvat, Robert J.

PA Searle, G. D., and Co., USA

SO U.S., 15 pp.

CODEN: USXXAM

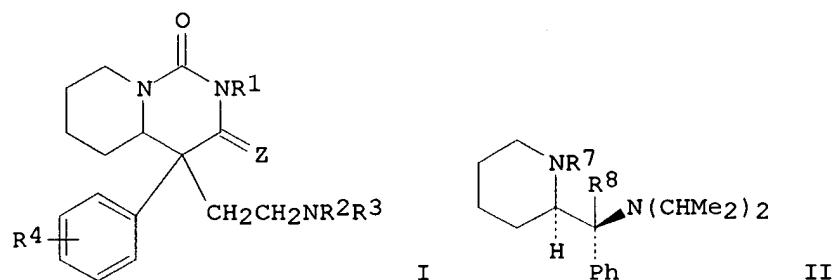
PI US 4680295 A 870714

AI US 86-825723 860203

DT Patent

LA English

GI



AB The title compds. [I; R₁ = H, C1-6 alkyl, C2-6 hydroxyalkyl, R₅CH₂, R₅CH₂CH₂; R₂, R₃ = C1-6 alkyl; R₄ = H, C1-6 alkyl, C1-6 alkoxy, Ph, halo; R₅ = C5-8 cycloalkyl, (un)substituted Ph; Z = O, R₆N; R₆ = H, C2-6 alkanoyl], their tautomeric forms and their pharmaceutically acceptable salts were prepd. as cardiac antiarrhythmics. (.+-.)-2-Phenyl-2-(2-pyridimyl)-4-[bis(1-methylethyl)amino]butanamide was hydrogenated over PtO₂ to give piperidine (.+-.)-II (R₇ = H, R₈ = CONH₂). This was treated with COCl₂ to give (.+-.)-II (R₇ = COCl, R₈ = cyano). The latter was amidated with 3,4-(MeO)₂C₆H₃CH₂CH₂NH₂ and the product cyclized by refluxing with NaH in THF to give, after acidification, trans-I.2HCl [R₁ = 3,4-(MeO)₂C₆H₃CH₂CH₂, R₂ = R₃ = Me₂CH, R₄ = H, Z = NH] (III). In dogs III inhibited ventricular arrhythmia induced coronary artery ligation with a min. ED of 1.2 mg/kg i.v.

L6 ANSWER 4 OF 9 CA COPYRIGHT 1998 ACS

AN 106:213973 CA

TI Process and intermediates for antiarrhythmic 1,3-diazabicyclo[4.4.0]dec-2-en-4-ones

IN McLaughlin, Kathleen Therese; Chorvat, Robert John; Prodan, Kathleen Ann

PA Searle, G. D., and Co., USA

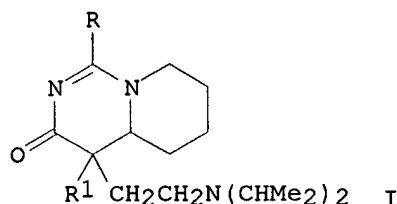
SO Eur. Pat. Appl., 33 pp.

CODEN: EPXXDW

PI EP 215477 A1 870325

DS R: BE, CH, DE, FR, GB, IT, LI, NL, SE

AI EP 86-112839 860917
PRAI US 85-777661 850919
DT Patent
LA English
GI



AB The title compds. (4,4a,5,6,7,8-tetrahydro-3H-pyrido[1,2-c]pyrimidin-3-ones) [I; R = H, alkyl, (un)substituted Ph; R1 = (un)substituted Ph] were prepd. as antiarrhythmics (no data) in improved yield and purity. (.+-.)-(Me2CH)2NCH2CH2CQPhCONH2 (II, Q = 2-pyridinyl) was catalytically hydrogenated to give diastereomeric (.+-.)-II (Q = 2-piperidinyl) which was acylated with Ac2O to give (.+-.)-II (Q = 1-acetyl-2-piperidinyl). The latter was stirred 2 h at room temp. with powd. KOH in Me2SO to give (.+-.)-I (R = Me, R1 = Ph).

L6 ANSWER 5 OF 9 CA COPYRIGHT 1998 ACS

AN 106:113135 CA

TI Molecular and structural basis of resting and use-dependent block of sodium current defined using disopyramide analogs

AU Yeh, J. Z.; TenEick, Robert E.

CS Dep. Pharmacol., Northwest. Univ., Chicago, IL, 60611, USA

SO Biophys. J. (1987), 51(1), 123-35

CODEN: BIOJAU; ISSN: 0006-3495

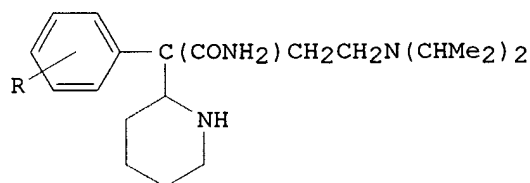
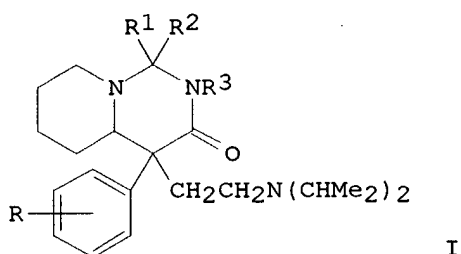
DT Journal

LA English

AB The effects of Norpace (disopyramide) [22059-60-5] and 14 closely-related structural analogs on the Na current of voltage clamped squid axons were examd. to det. which physico-chem. properties and which changes in the structure of the Norpace mol. can alter the nature of its Na channel-blocking actions. For both mono-tertiary and bis-tertiary amines, the potency to produce use-dependent block was proportional to mol. wt., whereas the correlation between potency to produce resting block and mol. wt. was significant only for bis-tertiary amines. The mono-compds. were more potent than the bis-compds. However, comparisons between compds. having similar mol. wts. and/or pKa values indicate that other factors also can influence blocking potency. For compds. within each homologous mono- or bis-tertiary amine series, hydrophobicity was found to influence the potency to produce use-dependent block of Na current. Use-dependent block was extant in axons internally exposed to pronase to remove the inactivation process, which indicates that inactivation is not an obligate condition for development of use-dependent block of Na current. An important role for the activation process in the development of use-dependent block of Na current is suggested by the finding that, in general, the voltage dependence of Na current activation paralleled that of use-dependent block. However, the potential dependence of use-dependent block produced by less hydrophobic but not by more hydrophobic compds. was shifted in the hyperpolarizing direction by removing Na from the external soln. Compds. with intermediate hydrophobicities altered the time course of Na current during its activating and inactivating phases. This finding can be explained by the kinetics of assocn. and disocn. of drug mols. with channel receptor sites during the development and relaxation of

use-dependent block rather than by postulating any major effect of drug to alter channel gating kinetics. The implications of the findings with respect to several factors believed to influence drug potency for resting and use-dependent block of the Na current in squid axon are examd. and discussed.

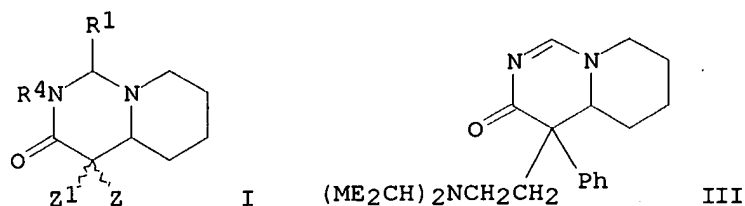
L6 ANSWER 6 OF 9 CA COPYRIGHT 1998 ACS
AN 103:104918 CA
TI Synthesis and structure-activity relationships of a new series of antiarrhythmic agents: 4,4-disubstituted hexahydro-3H-pyrido[1,2-c]pyrimidin-3-ones and related compounds
AU Chorvat, Robert J.; Prodan, Kathleen A.; Adelstein, Gilbert W.; Rydzewski, Robert M.; McLaughlin, Kathleen T.; Stamm, Margarete H.; Frederick, Leo G.; Schniepp, Henry C.; Stickney, Janice L.
CS Dep. Med. Chem., G. D. Searle Co., Skokie, IL, 60077, USA
SO J. Med. Chem. (1985), 28(9), 1285-91
CODEN: JMCMAR; ISSN: 0022-2623
DT Journal
LA English
OS CASREACT 103:104918; CJACS
GI



AB Pyrido[1,2-c]pyrimidin-3-ones I (R = H, 2-Cl, 4-Ph; R1 = H, Me, Et, Ph; R2 = R3 = H; R2R3 = bond) were prepd. from butanamides II. Individual racemates of II were converted to pure racemic I that were evaluated for antiarrhythmic and anticholinergic activity. Selected I were evaluated for hemodynamic effects in dogs. I (R = H, R1 = Me, R2 = R3 = H, R2R3 = bond) showed the most favorable pharmacol. profiles. I (R = H, R1 = Me, R2R3 = bond) was chosen for toxicity testing because of its lack of noncompetitive inhibition of acetylcholine-induced contractions of guinea pig ileum.

L6 ANSWER 7 OF 9 CA COPYRIGHT 1998 ACS
AN 101:90961 CA
TI Pyridopyrimidinones
IN Adelstein, Gilbert William; Chorvat, Robert John
PA Searle, G. D., and Co., USA
SO Eur. Pat. Appl., 35 pp.
CODEN: EPXXDW
PI EP 104647 A2 840404
DS R: BE, CH, DE, FR, GB, IT, LI, NL, SE
AI EP 83-109607 830927

PRAI US 82-424936 820927
 US 83-532120 830914
 DT Patent
 LA English
 GI



AB The title compds. I [R₁ = H, C1-6 alkyl, Ph; Z = H, (CH₂)_nNR₂R₃ (R₂, R₃ = C1-6 alkyl, R₂R₃ = C4-6 alkylene, n = 1-6); Z₁ = H, (un)substituted Ph; R₄ = H, C1-6 alkyl], useful as antiarrhythmics with reduced anticholinergic activity (no data), were prepd. Thus, 105.3 g (Me₂CH)₂NCH₂CH₂CRPhCONH₂ (II, R = 2-pyridyl) was hydrogenated over PtO₂ to give 26.0 g II (R = 2-piperidinyl) which (16.35 g) was condensed with HC(OMe)₂NMe₂ to give 12.9 g III. Hydrogenation of 14.0 g III over Pd/C gave 1.0 g I [R₁ = R₄ = H, Z = Ph, Z₁ = (Me₂CH)₂NCH₂CH₂].

L6 ANSWER 8 OF 9 CA COPYRIGHT 1998 ACS

AN 79:126273 CA

TI Syntheses of thioacetamide derivatives. I

AU Sanno, Yasushi; Kanai, Yoshio; Nohara, Akira; Hirata, Takeo

CS Takeda Chem. Ind., Ltd., Osaka, Japan

SO Takeda Kenkyusho Ho (1973), 32(2), 125-9

CODEN: TAKHAA

DT Journal

LA Japanese

AB In order to find a new nonanticholinergic antiulcer agent, 2-phenyl-2-[2-(N-methylpyridinium)][thioacetamide iodide, 2-phenyl-2-(2-piperidyl)thioacetamide, 2-phenyl-2-(3-pyridyl)thioacetamide, 1-methyl-4-(2-pyridyl)piperidine-4-thioformamide, DL-phenylalaninethioamide, 2-[2-(1-benzylimidazolyl)]thioacetamide, and 3-(2-pyridyl)thiopropionamide were prepd.

L6 ANSWER 9 OF 9 CA COPYRIGHT 1998 ACS

AN 69:77026 CA

TI Condensed 2-azetidinones. II. Isomeric 3-phenyl-1-azabicyclo[4,2,0]octan-2-ones

AU Moll, F.

CS Univ. Tuebingen, Tuebingen, Ger.

SO Arch. Pharm. (Weinheim) (1968), 301(4), 250-62

CODEN: APBDAJ

DT Journal

LA German

AB .alpha.-Phenyl-.alpha.-pyrid-2-ylacetonitrile was converted into the corresponding amide, and reduced to .alpha.-phenyl-.alpha.-piperid-2-ylacetamide, which was treated with 18% HCl 6 hrs. at 110.degree. to give .alpha.-phenyl-.alpha.-piperid-2-ylacetic acid-HCl (I). Attempts to remove the HCl from I gave mainly piperidylacetic acid. .alpha.-Ethyl-.alpha.-phenyl-.alpha.-piperid-2-ylacetamide was similarly obtained, but could not be converted into .alpha.-ethyl-.alpha.-phenyl-.alpha.-piperid-2-ylacetic acid-HCl. .alpha.-Phenyl-.alpha.-piperid-2-ylacetyl chloride-HCl (II) was prepd. from .alpha.-phenyl-.alpha.-piperid-2-ylacetic acid and SOCl₂. II was treated with Et₃N 24 hrs. at room temp. to give

3-phenyl-1-azabicyclo[4.2.0]octan-2-one (III), also obtained by treating I with dicyclohexylcarbodiimide. The isomers of II were sepd. by gas chromatog. on polyethylene glycol (20,000) at 220.degree., 106 ml./min. He. The cis isomer, m. 70.degree., had a retention time of 20 min. and the trans isomer 23 min. III was also prepd. by photolysis of phenylglyoxylpiperidine dianzo ketone as described by E. J. Corey and A. M. Felix (1965). The trans isomer was the major product in this reaction.

AN 1968:108272 CAPLUS

DN 68:108272

TI Nature of aprotic acidity in aluminosilicate catalysts

AU Uridiya, L. Ya.; Mdivnishvili, O. M.

SO Tr. Kavk. Inst. Miner. Syr'ya (1965), No. 6, 43-6

CODEN: TKMMA3

DT Journal

LA Russian

AB While detg. the aprotic centers in clays of various mineralogical compn. the authors advanced a hypothesis that the protons present on the surface of aluminosilicates posses under certain conditions the properties of aprotic centers. The proton similarly to the aprotic **Lewis acids** is characterized by a strong affinity towards an electron pair. Thus, the aprotic **acidity** was detd. of substances which are **Lewis acids**, Al_2O_3 , $Al_2(SO_4)_3$, and $AlCl_3$ and of protonic **acids**, 36% HCl and 96% H_2SO_4 . In the titrn. of the aprotic **acid**, C_6H_6 was used as a solvent. The titrn. in all cases was carried out by Et acetate. Thus, 96% H_2SO_4 was found to react actively with a **Lewis base**, AcOEt. This interaction takes place in concd. H_2SO_4 only when the ionization and cond. tend to zero and when there is no ionization of protons. The assocn. with AcOEt is presumed to take place by a **hydrogen bond**. In natural aluminosilicates, on a dehydrated surface, the presence of protons is possible which can give analogous compds. via H bridges. Thus, the aprotic **acidity** due to triply coordinated Al atoms surrounded by silica tetrahedra is not the only source of the aprotic centers in aluminosilicates.

=> s chiral(3a)resolv? (3a)agent?

50359 CHIRAL
102243 RESOLV?
719245 AGENT?

L1 60 CHIRAL(3A)RESOLV? (3A)AGENT?

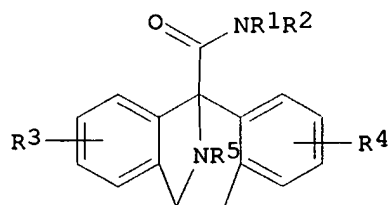
=> s l1 and (tartar? or malic or camphor? or glucoron?)

15234 TARTAR?
14578 MALIC
9171 CAMPHOR?
321 GLUCORON?

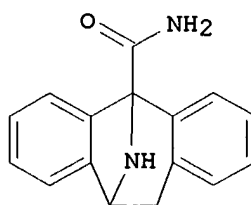
L2 8 L1 AND (TARTAR? OR MALIC OR CAMPHOR? OR GLUCORON?)

=> d bib abs 1-8

L2 ANSWER 1 OF 8 CA COPYRIGHT 1998 ACS
AN 126:8007 CA
TI Preparation of chiral 5-aminocarbonyl-5H-dibenzo[a,d]cyclohepten-
5,10-imines by optical resolution
IN Jones, Tappey H.; Rice, Kenner C.
PA United States Dept. of Health and Human Services, USA; Rice, Kenner,
C.; Neurogen Corporation
SO PCT Int. Appl., 16 pp.
CODEN: PIXXD2
PI WO 9632390 A1 961017
DS W: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE,
ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LT, LU,
LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG,
SI, SK
RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, DE, DK, ES, FI, FR, GA, GB,
GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG
AI WO 96-US4906 960410
PRAI US 95-420013 950411
DT Patent
LA English
OS MARPAT 126:8007
GI



I



II

AB Processes for resolving a racemic mixt. of 5-aminocarbonyl-5H-

dibenzo[a,d]cyclohepten-5,10-imines [I; R1, R2 = H, alk(en/yn)yl, cycloalk(en)yl, or R1R2 forms ring; R3, R4 = H, alk(en/yn)yl, OH, (alkyl)amino, alkoxy, cyano, NO2, haloalkyl, SH; R5 = H, alk(en/yn)yl, OH, Ph, haloalkyl, aminoalkyl, phenylmethyl, alkoxy; or R1R5 forms ring; all groups may be further substituted] into the component enantiomers are disclosed. In particular, II is resolved by recrystn. of diastereomeric tartrate salts. I are known anticonvulsants (no data). For example (+-)-II (prepn. given) was treated with L-(+)-**tartaric** acid [L-(+)-III] in aq. EtOH, and the soln. was boiled, filtered, and crystd. twice to give the diastereomeric salt (5S,10R)-(-)-II.L-(+)-III. Treatment of the salt with NH4OH, extn. into CH2Cl2, and recrystn. from aq. EtOH gave (5S,10R)-(-)-II. Similar treatment of the above crystn. filtrates with D-(-)-III gave (5R,10S)-(+)-II. Diastereomeric carbamoylation with (S)-(+)-naphthylethyl isocyanate showed that the product had an enantiomeric excess (ee) of 96%.

L2 ANSWER 2 OF 8 CA COPYRIGHT 1998 ACS
 AN 124:175586 CA
 TI Preparation of 1-phenyl-3-butyne derivatives as (S)-2-amino-1-phenyl-3-butene precursors
 IN Onomura, Osamu; Ueda, Yoichiro; Murai, Yoshuki
 PA Daicel Chem, Japan
 SO Jpn. Kokai Tokkyo Koho, 16 pp.
 CODEN: JKXXAF
 PI JP 07247246 A2 950926 Heisei
 AI JP 94-65634 940309
 DT Patent
 LA Japanese
 AB 2-Methanesulfonyloxy-1-phenyl-3-butyne (I) is prepd. 2-Amino-1-phenyl-3-butyne (II) is prepd. by treating I with NH3 sources. II is purified by prepn. of II acid salts. (S)-II.L-**tartaric** acid (III) salt is prepd. by treating racemic II with L-III, then crystg. the (S)-II.L-III. (S)-II is prepd. by hydrolysis of (S)-II.L-III. (R)-II.D-III is prepd. by treating racemic II with D-III, then crystg. the (R)-II.D-III. (R)-II is prepd. by hydrolysis of (R)-II.D-III. A soln. of 296.6 g 2-hydroxy-1-phenyl-3-butyne and Et3N in CH2Cl2 was treated dropwise with 244.0 g MeSO2Cl at 5-10.degree. over 2 h, then treated for 30 min to give 495.5 g I, which was treated with Me2CHOH and 40.98 mol aq. NH3 at 65.degree. for 9 h, then mixed with HCl under ice cooling to give 277.0 g II.HCl. The II.HCl was treated with aq. NaOH to give 176.0 g II, which was treated dropwise into a soln. of L-III in MeOH over 30 min, then settled at room temp. over night to give 60% (+)-(S)-II.L-III of .gtoreq.98% e.e. The (+)-(S)-II.L-III (39.45 g) was treated with aq. NaOH to give 19.9 g (S)-II of .gtoreq.97% e.e.

L2 ANSWER 3 OF 8 CA COPYRIGHT 1998 ACS
 AN 118:212651 CA
 TI Separation of a racemic mixture of enantiomers of optically active molecules or ions. Resolution of (+-)-fenfluramine.
 IN Bouaziz, Roger; Legrand-Mofaadel, Nadine
 PA Universite de Rouen, Fr.
 SO Fr. Demande, 14 pp.
 CODEN: FRXXBL
 PI FR 2672285 A1 920807
 AI FR 91-1513 910205
 DT Patent
 LA French
 AB A process is given for the resoln. of racemates of chiral mols. or ions by addn. of a chiral salification agent and fractional crystn. of the less sol. diastereomeric salt. The **chiral resolving agent** is partially substituted, in proportions defined by examn. of the equil. between phases, by a non-chiral agent with the same characteristics, such as a mineral or

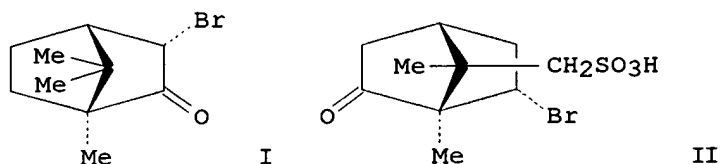
org. base or acid, and the amt. of org. solvent is quantified and removed from the reaction mixt. by isothermal evapn. The process is described for the resoln. of (.+-.)-fenfluramine with (+)-**camphoric** acid and HCl in EtOH, leading to the isolation of the (+)-**camphorate** of (+)-(S)-fenfluramine.

L2 ANSWER 4 OF 8 CA COPYRIGHT 1998 ACS
AN 118:124183 CA
TI Process for the selection of a resolving agent suitable for separating enantiomers and process for determining the enantiomer separation attainable by means of the resolving agent
IN Acs, Maria; Fogassy, Elemer; Gal, Sandor; Kozma, David; Pokol, Gyorgy
PA Budapesti Muszaki Egyetem, Hung.
SO Hung. Teljes, 7 pp.
CODEN: HUXXB
PI HU 60706 A2 921028
AI HU 91-1171 910411
DT Patent
LA Hungarian
AB Optically active base or acid resolving agents for racemic acids or bases are identified as those affording 1:1 diastereomeric salt mixts. whose DSC traces display two peaks. A formula for theor. attainable enantiomer sepn. efficiency (by fractional crystn.) in terms of DSC data is presented: $[(1-2x)/(2-2x)] \ln 2x = (H_2/R)[(1/T_2)-(1/T_1)]$, where T_i = DSC peak min., H_2 = heat of fusion corresponding to the second peak, R = universal gas const., and S (the product of enantiomer yield and optical purity in fractional crystn.) = $(1-2x)/(1-x)$. Thus, the DSC data for a diastereomeric salt mixt. prepd. from 0.115 g racemic .alpha.-(6-methoxy-2-naphthyl)-.alpha.-methylacetic acid and 0.098 g D-(-)-N-methylglucamine ($T_1 = 398$ K, $T_2 = 414$ K, $H_2 = 37.2$ kJ/mol) predicted enantiomer sepn. of 0.65; fractional crystn. of the diastereomeric salts led to an optimal enantiomer sepn. of 0.65.

L2 ANSWER 5 OF 8 CA COPYRIGHT 1998 ACS
AN 118:101242 CA
TI Process for resolving a racemic composition
IN Acs, Maria; Fogassy, Elemer; Szili, Tímea
PA Budapest Muszaki Egyetem, Hung.
SO Hung. Teljes
CODEN: HUXXB
PI HU 60227 A2 920828
AI HU 91-661 910227
DT Patent
LA Hungarian
AB Enantiomeric mixts. of N-contg. bases are prepd. by resoln. of the racemates in a process involving mixing of the racemic N-contg. base with less than 1 equiv optically active O-acylated **tartaric** acid (in this particular case), permitting the mixt. to stand, and then at elevated temp. (reduced pressure if necessary) condensing the resultant vapors. Thus, 2.4 g racemic .alpha.-methylbenzylamine and 0.75 g L-(+)-mandelic acid (0.02 and 0.005 mol, resp.) are mixed and allowed to stand for 30 min. By means of external heating, this mixt. is then distd. at 0.02 bar (vapor temp. 30.degree.), with sudden decrease of vapor temp. marking the end of distn.; 1.1 g material is collected with $[\alpha]_{D20} = +1.6$.degree.. If 1.2 g racemic .alpha.-methylbenzylamine is used, all else as above, then 0.3 g material is collected with $[\alpha]_{D20} = 6.6$.degree.. Amplification of sp. rotation is achieved by repetition of the distn. procedure with optically active distillate.

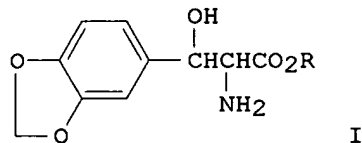
L2 ANSWER 6 OF 8 CA COPYRIGHT 1998 ACS
AN 110:8422 CA
TI Synthesis, structure and absolute configuration of a new

AU Degnbol, Johnny; Hammershoei, Anders
 CS Chem. Dep., Univ. Copenhagen, Copenhagen, DK-2100, Den.
 SO Acta Chem. Scand., Ser. B (1988), B42(6), 390-6
 CODEN: ACBOCV; ISSN: 0302-4369
 DT Journal
 LA English
 GI



AB Treatment of (+)-(1R)-3-endo-bromobornan-2-one [(+)-.alpha.-bromocamphor] (I) in fuming H₂SO₄ (34% SO₃) gave (-)-(1S)-6-endo-bromo-2-oxobornane-8-sulfonic acid (II) in a facile synthesis. The structure and abs. configuration of the ammonium salt was detd. by x-ray diffraction (orthorhombic, P2₁2₁2₁, a = 7.216(5), b = 10.793(3), c = 16.362(4) .ANG.; Z = 4). Under these conditions, sulfonation is accompanied by extensive rearrangement of the carbon skeleton amounting formally to inversion of the **camphor** segment. The structural changes are rationalized in terms of carbocation rearrangements consistent with results of other work relating to the racemization of **camphor** in H₂SO₄. The new isomer reported here is suggested as a member in the arsenal of easily-produced **chiral resolving agents**.

L2 ANSWER 7 OF 8 CA COPYRIGHT 1998 ACS
 AN 107:97119 CA
 TI Optically active threo-3-(3,4-methylenedioxyphenyl) serine
 IN Ishizumi, Kikuo; Maeshima, Kaoru; Nagata, Shoji; Kojima, Atsuyuki
 PA Sumitomo Pharmaceuticals Co., Ltd., Japan
 SO Eur. Pat. Appl., 9 pp.
 CODEN: EPXXDW
 PI EP 204481 A2 861210
 DS R: AT, BE, CH, DE, FR, GB, IT, LI, NL, SE
 AI EP 86-303979 860527
 PRAI JP 85-115588 850529
 DT Patent
 LA English
 GI



AB The title optically active compd. (I; R = H), useful as an intermediate for optically active threo-3-(3,4-dihydroxyphenyl)serine, was obtained by optical resolu. of racemic I (R = alkyl) with a **resolving agent** selected from **chiral** pyroglutamic acid or chiral dibenzoyltartaric acid. Thus, 0.50 g DL-threo-I (R = Me) and 0.79 g dibenzoyl-L-**tartaric** acid monohydrate were dissolved in 5 mL MeOH and

the soln. was left standing at 0-5.degree. for 72 h to give L-threo-I (R = Me) dibenzoyl-L-tartrate salt which was converted to the free base and saponified to give L-I (R = H).

L2 ANSWER 8 OF 8 CA COPYRIGHT 1998 ACS

AN 106:66522 CA

TI Enantioselectivity of hydrogen-bond association in liquid-solid chromatography

AU Dobashi, Akira; Dobashi, Yasuo; Hara, Shoji

CS Tokyo Coll. Pharm., Tokyo, 192-03, Japan

SO J. Liq. Chromatogr. (1986), 9(2-3), 243-67

CODEN: JLCHD8; ISSN: 0148-3919

DT Journal

LA English

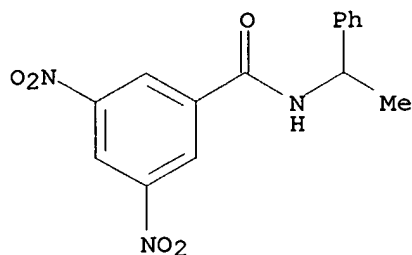
AB Liq. chromatog. resolsns. of enantiomers induced by mol. assocn. whose main driving force is the action of weak H bonds are described. The H bond assocn. potential was first demonstrated through the optical resoln. of racemic N-acylated amino acid esters using a chiral stationary phase (CSP) (N-acyl-L-valylamino)propyl silica gel. Following this preliminary study, application was made of the chiral mobile chiral graft of CSP is reproduced, to the resoln. of the above solute enantiomers in liq.-solid chromatog. The addn. of N-acetyl-L-valine tert-butylamide to the nonaq. mobile phase solvent of a silica gel column successfully brought about this optical resoln.; by this method, a novel and more effective **chiral resolving agent** was found. Two types of chiral additives derived from a chiral skeleton (R,R)-**tartaric** acid were found capable of resolving various kinds of enantiomers, such as dialkyl tartrate and dialkyl tartramide. Of these two, the latter in particular, having an iso-Pr substituent, led to a wide range of resoln. of enantiomers of the following categories: .alpha.- and .beta.-hydroxy carboxylic acid, .beta.-hydroxy ketone, .beta.-amino alc., .alpha.-amino acid, .alpha.-hydroxy ketoxime derivs., and bi-.beta.-naphthol. This occurred when the enantiomers, except .beta.-hydroxy ketones, .alpha.-hydroxy ketoximes, 1,2-diols and bi-.beta.-naphthol, were derivatized so as to respond to the H bonding sites of the additive mols.

RL: PUR (Purification or recovery); RCT (Reactant); PREP
(Preparation)

(optical resoln. with support-bound **tartaric** acid-amino
acid monoamide derivs. as reagents for chirality recognition and
chromatog. **resolving** agents)

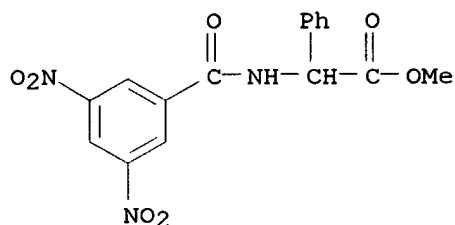
RN 14402-00-7 CA

CN Benzamide, 3,5-dinitro-N-(1-phenylethyl)- (9CI) (CA INDEX NAME)



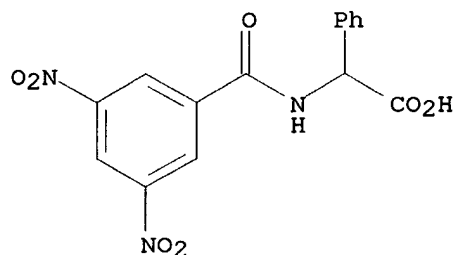
RN 74928-23-7 CA

CN Benzeneacetic acid, .alpha.-[(3,5-dinitrobenzoyl)amino]-, methyl
ester (9CI) (CA INDEX NAME)



RN 74958-71-7 CA

CN Benzeneacetic acid, .alpha.-[(3,5-dinitrobenzoyl)amino]- (9CI) (CA
INDEX NAME)



IT 69632-31-1P 69632-32-2P 69632-49-1P,

N-3,5-Dinitrobenzoyl-L-phenylglycine methyl ester

69632-50-4P, N-3,5-Dinitrobenzoyl-D-phenylglycine methyl

ester 74927-72-3P, N-3,5-Dinitrobenzoyl-D-phenylglycine

90761-62-9P, N-3,5-Dinitrobenzoyl-L-phenylglycine

RL: PUR (Purification or recovery); SPN (Synthetic preparation);

PREP (Preparation)

(prepn. by chromatog. optical resoln. with support-bound

tartaric acid-amino acid monoamide derivs. as reagents

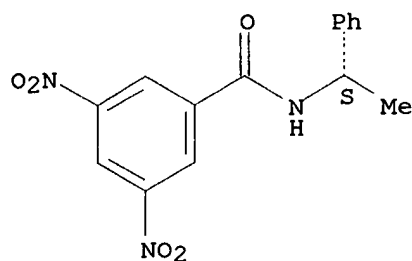
for chirality recognition and chromatog. **resolving**

agents)

RN 69632-31-1 CA

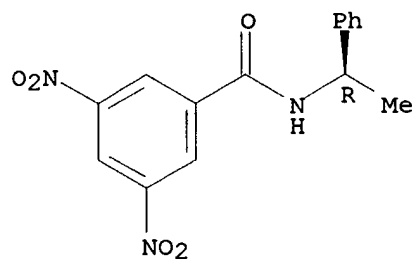
CN Benzamide, 3,5-dinitro-N-(1-phenylethyl)-, (S)- (9CI) (CA INDEX
NAME)

Absolute stereochemistry.



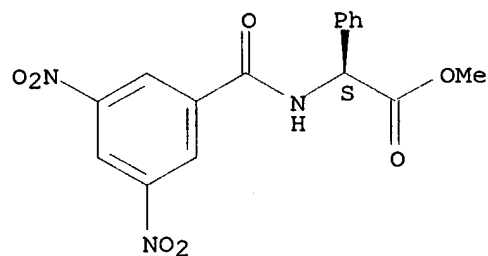
RN 69632-32-2 CA
CN Benzamide, 3,5-dinitro-N-(1-phenylethyl)-, (R)- (9CI) (CA INDEX
NAME)

Absolute stereochemistry.



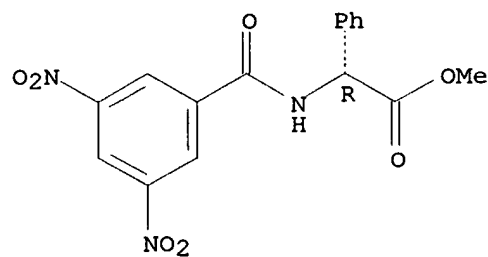
RN 69632-49-1 CA
CN Benzeneacetic acid, .alpha.-[(3,5-dinitrobenzoyl)amino]-, methyl
ester, (S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



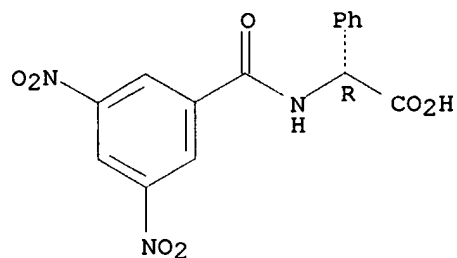
RN 69632-50-4 CA
CN Benzeneacetic acid, .alpha.-[(3,5-dinitrobenzoyl)amino]-, methyl
ester, (R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



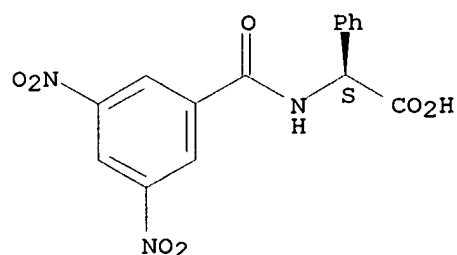
RN 74927-72-3 CA
 CN Benzeneacetic acid, .alpha.-[(3,5-dinitrobenzoyl)amino]-, (R)- (9CI)
 (CA INDEX NAME)

Absolute stereochemistry.

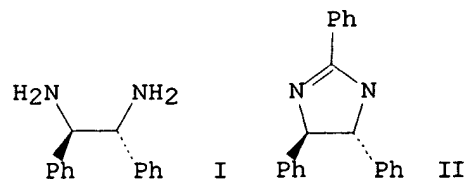


RN 90761-62-9 CA
 CN Benzeneacetic acid, .alpha.-[(3,5-dinitrobenzoyl)amino]-, (S)- (9CI)
 (CA INDEX NAME)

Absolute stereochemistry.



L9 ANSWER 2 OF 2 CA COPYRIGHT 1998 ACS
 AN 120:163554 CA
 TI Improved synthesis and resolution of (.+-.)-1,2-diphenylethanediamine
 AU Chen, Weiping; Yang, Fuqiu
 CS Shanghai Inst. Pharm. Ind., Shanghai, 200437, Peop. Rep. China
 SO Zhongguo Yiyao Gongye Zazhi (1993), 24(6), 280-1
 CODEN: ZYGZEA; ISSN: 1001-8255
 DT Journal
 LA Chinese
 OS CASREACT 120:163554
 GI



AB The title compd. (I) is prepd. via reaction of benzaldehyde with NH₃, heating the resulting PhCH:NCHPhN:CHPh in EtOH contg. NaOH, heating the resulting imidazoline II with NaOAc-Ac₂O, and heating the resulting (.+-.)-Bz-NHCHPhCHPhNHAc with HBr in HOAc. I is **resolved** via forming diastereomeric salts with (+)-**tartaric acid** and decomp. the isolated (-)-I.(+)-tartrate.

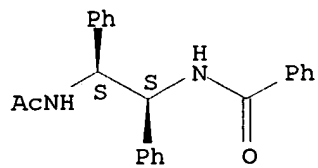
IT 69576-65-4P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and deacylation of)

RN 69576-65-4 CA

CN Benzamide, N-[2-(acetylamino)-1,2-diphenylethyl]-, (R*,R*)- (9CI)
(CA INDEX NAME)

Relative stereochemistry.



=> d bib abs hitstr 1-2

L9 ANSWER 1 OF 2 CA COPYRIGHT 1998 ACS
 AN 123:144624 CA
 TI Preparation of support-bound **tartaric** acid-amino acid
 monoamide derivative as reagent for chirality recognition and
resolving agent for chromatography
 IN Ooi, Takafumi; Kitahara, Hajime; Matsushita, Yasuhiro; Kisu, Naoko
 PA Sumika Bunseki Center Kk, Japan
 SO Jpn. Kokai Tokkyo Koho, 11 pp.
 CODEN: JKXXAF
 PI JP 06298672 A2 941025 Heisei
 AI JP 93-89956 930416
 DT Patent
 LA Japanese
 AB A reagent for chirality recognition has a structure comprising
 N-(3-carboxy-2,3-dihydroxypropionyl)amino acid N-(silylalkyl)amide
 (organosilane compd.) R1R2R3Si-R4-NHCO-R5-NR6COCH(OH)CH(OH)CO2H [R1,
 R2, R3 = alkyl, alkoxy, OH, halo, provided that at least one of R1 -
 R3 = alkoxy or halo; R4 = lower alkylene; R5 = residue derived by
 removing one NH2 and one CO2H group from optically active L- or
 D-amino acid; R6 = H, lower alkyl; or R5R6 = residue derived by
 removing one imino and one CO2H group from optically active L- or
 D-imino acid, provided that the remaining OH, NH, and NH2 in R5 may
 be substituted by Y-Z (wherein Y = CO or CONH and Z = lower alkyl,
 mono- or bicyclic aryl or aralkyl); each portion of **tartaric**
 acid, amino acid, and imino acid is in an optically active form]
 grafted to an inorg. support having hydroxy groups on the surface.
 A chromatog. packing material comprises the compd. described above.
 This chiral recognition reagent is durable due to chem. stability,
 has in the structure both a ligand exchange part (which enables
 direct sepn. of optical isomers such as amino acids and oxyacids)
 and a hydrogen-bonding interaction part (which enable sepn. of
 optical isomer derivs. such as amines, amino acids, and carboxylic
 acids), and are useful for optical resoln. of racemates. Thus, 200
 g silica gel (av. grain diam. 5 .mu.m, av. pore diam. 120.ANG., and
 surface area 330 m2/g) was dried at 120.degree. in vacuo for 2 h and
 refluxed with 200 g 3-aminopropyltriethoxysilane in 1 L dry THF for
 3 h to give 3-aminopropylated silica gel (0.94 mmol 3-aminopropyl
 group/1 g) which was condensed with Boc-Val-OH and after removing
 the Boc group, with 2,3-di-O-acetyl-L-**tartaric** acid
 followed by capping the residual amino group with Ac2O and
 hydrolysis with HClO4 in refluxing aq. MeCN to give
 N-(N-3-carboxy-2,3-dihydroxypropionyl-L-valyl)-3-aminopropylsilyl-
 grafted silica gel as a chromatog. packing material. A stainless
 steel column (inner diam. 4 mm .times. length 25 cm) packed with the
 latter packing material **resolved** racemic alcs.
 (binaphthol, uniconazole, and diniconazole), racemic amino acid
 deriv. [Ac-Val-OEt, DNB-Val-OMe (DNB = 3,5-dinitrobenzoyl),
 Ac-Phe-OMe, N-acetylphenylglycine, and N-DNB-phenylglycine], and
 racemic 1,1'-binaphthyl-2,2'-diyl hydrogen phosphate with sepn.
 coefficient (.alpha. = K1'/K2', wherein K1', K2' = retention
 coefficient for each enantiomer) of 1.037-1.158.
 IT 14402-00-7P 74928-23-7P 74958-71-7P,
 N-3,5-Dinitrobenzoyl-DL-phenylglycine

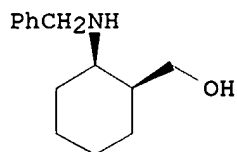
=> s tartaric and mandelic and dibenzoyl

14389 TARTARIC
2744 MANDELIC
1992 DIBENZOYL
L1 5 TARTARIC AND MANDELIC AND DIBENZOYL

=> d bib abs 1-5

L1 ANSWER 1 OF 5 CA COPYRIGHT 1998 ACS
AN 123:328858 CA
TI Separation of Diastereomers by Capillary Zone Electrophoresis with
Polymer Additives: Effect of Polymer Type and Chain Length
AU Schuetzner, Wolfgang; Fanali, Salvatore; Rizzi, Andreas; Kenndler,
Ernst
CS Istituto di Cromatografia, CNR, Monterotondo Scalo, 00016, Italy
SO Anal. Chem. (1995), 67(21), 3866-70
CODEN: ANCHAM; ISSN: 0003-2700
DT Journal
LA English
OS CJACS-IMAGE; CJACS
AB Diastereomeric derivs. of enantiomers are sepd. by capillary zone
electrophoresis in nonchiral sepn. systems in the presence of linear
polymers. These polymers significantly influence the mobilities of
the analytes as well as the stereoselectivity of the system. Three
types of linear polymers, poly(vinylpyrrolidone),
poly(ethyleneglycol), and poly(acrylamide), are studied to det.
their influence on the stereoselective sepn. of diastereomeric
derivs. of .alpha.-amino acids obtained by reaction with optically
pure (+)-O,O'-**dibenzoyl-L-tartaric** anhydride.
Differences are found in the strength of the polymer effect and the
effected migration order. Polymer chain length had no impact on
stereoselectivity.

L1 ANSWER 2 OF 5 CA COPYRIGHT 1998 ACS
AN 118:101538 CA
TI A convenient synthesis of cis-(1R)-N-benzyl-2S)-
(hydroxymethyl)cyclohexylamine
AU Vanderplas, B.; Murtiashaw, C. W.; Sinay, T.; Urban, F. J.
CS Cent. Res. Div., Pfizer Inc., Groton, CT, 06340, USA
SO Org. Prep. Proced. Int. (1992), 24(6), 685-7
CODEN: OPPIAK; ISSN: 0030-4948
DT Journal
LA English
OS CASREACT 118:101538
GI



I

AB A convenient synthesis of the title compd. I is described. Reductive amination of com. available Et 2-cyclohexanonecarboxylate with PhCH₂NH₂ contg. Na(OAc)₃BH in CH₂Cl₂ gave (.+-.)-cis-N-benzyl-2-(carboethoxy)cyclohexylamine (II). Redn. of ester II with Vitride followed by resoln. with (+)-**mandelic** acid gave optically active amino alc. I. The abs. configuration of I was confirmed by x-ray anal. of its **dibenzoyl-L-tartaric** acid salt.

L1 ANSWER 3 OF 5 CA COPYRIGHT 1998 ACS

AN 118:101242 CA

TI Process for resolving a racemic composition

IN Acs, Maria; Fogassy, Elemer; Szili, Timea

PA Budapest Muszaki Egyetem, Hung.

SO Hung. Teljes

CODEN: HUXXB

PI HU 60227 A2 920828

AI HU 91-661 910227

DT Patent

LA Hungarian

AB Enantiomeric mixts. of N-contg. bases are prepd. by resoln. of the racemates in a process involving mixing of the racemic N-contg. base with less than 1 equiv optically active O-acylated **tartaric** acid (in this particular case), permitting the mixt. to stand, and then at elevated temp. (reduced pressure if necessary) condensing the resultant vapors. Thus, 2.4 g racemic .alpha.-methylbenzylamine and 0.75 g L-(+)-**mandelic** acid (0.02 and 0.005 mol, resp.) are mixed and allowed to stand for 30 min. By means of external heating, this mixt. is then distd. at 0.02 bar (vapor temp. 30.degree.), with sudden decrease of vapor temp. marking the end of distn.; 1.1 g material is collected with [.alpha.]D₂₀ = +1.6.degree.. If 1.2 g racemic .alpha.-methylbenzylamine is used, all else as above, then 0.3 g material is collected with [.alpha.]D₂₀ = 6.6.degree.. Amplification of sp. rotation is achieved by repetition of the distn. procedure with optically active distillate.

L1 ANSWER 4 OF 5 CA COPYRIGHT 1998 ACS

AN 104:186157 CA

TI Acyl(amino)naphthalene derivatives

IN Ohashi, Naohito; Maejima, Kaoru; Ishizumi, Kikuo

PA Sumitomo Chemical Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 7 pp.

CODEN: JKXXAF

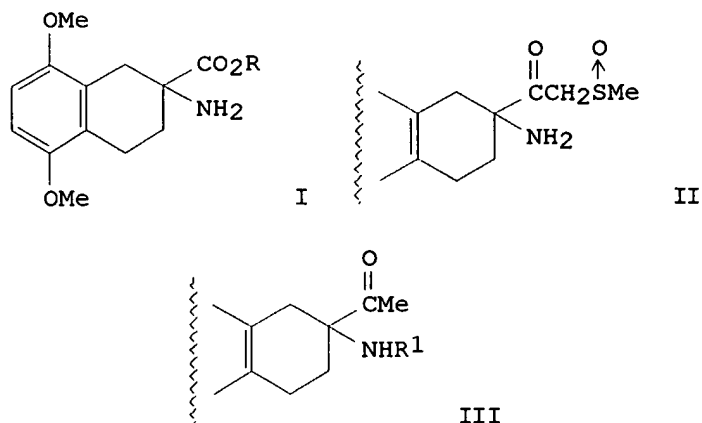
PI JP 60166650 A2 850829 Showa

AI JP 84-22487 840208

DT Patent

LA Japanese

GI



AB Racemic or optically active I (R = ester residue) were treated with methylsulfinyl carbanion to give II, which was desulfurated to give III (R₁ = H), which was acylated to give III (R₁ = acyl), useful as neoplasm inhibitors (no data). (.+-.)-I was treated with optically active **mandelic** acid, (**dibenzoyl**) **tartaric** acid, or (3-bromo)camphorsulfonic acid to give 1-I acid salts, and I acid diastereomer salts, which were resolved to 1-I acid salts and d-I acid salts, each of which was treated with a base to give optically active I. Thus, 9.53 g (.+-.)-I (R = Me) was treated with methylsulfonyl carbanion prep. from TMF and Me₂SO to give 10.88 g (.+-.)-II, which was treated with activated Zn powder at 65.degree. for 2 h, then treated with Ac₂O to give 8.42 g III (R = Ac).

L1 ANSWER 5 OF 5 CA COPYRIGHT 1998 ACS

AN 97:38738 CA

TI Enantiomeric .alpha.-aminopropiophenones (cathinone): preparation and investigation

AU Berrang, Bertold D.; Lewin, Anita H.; Carroll, F. Ivy

CS Chem. Life Sci. Group, Research Triangle Inst., Research Triangle Park, NC, 27709, USA

SO J. Org. Chem. (1982), 47(13), 2643-7

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA English

OS CJACS

AB (-)-PhCOCHMeNH₂ is a constituent of *Catha edulis*. Resoln. of (.+-.)-PhCOCHMeNH₂ with **mandelic** acid gave only minute yields and **tartaric** acid gave only somewhat better results. (.+-.)-Norephedrine was resolved in high yield into its (+) and (-) antipodes with O,O-**dibenzoyl**-d-**tartaric** acid and each enantiomer was converted into its N-formyl deriv. and oxidized with CrO₃ in pyridine. Hydrolysis with 20% HCl at 40.degree. gave optically pure PhCOCHMeNH₂.HCl without racemization. (-)-PhCOCHMeNH₂ was obtained in 39% and the (+) enantiomer in 40% overall yield from (.+-.)-norephedrine. The characterization of PhCOCHMeNH₂ and its salts and their stability in various solvents were discussed.

=> s dipivaloyl(2a)tartar?

80 DIPIVALOYL
15234 TARTAR?
L5 1 DIPIVALOYL(2A)TARTAR?

=> d bib abs

L5 ANSWER 1 OF 1 CA COPYRIGHT 1998 ACS
AN 93:168605 CA
TI Optically active .alpha.-amino acids and their derivatives
IN Duhamel, Lucette; Piaquevent, Jean Christophe
PA Agence Nationale de Valorisation de la Recherche, Fr.
SO Eur. Pat. Appl., 23 pp.
CODEN: EPXXDW
PI EP 7834 800206
PRAI FR 78-20345 780707
DT Patent
LA French
AB Optically active .alpha.-amino acids were prepd. by deprotonation
and enantioselective reprotonation of the racemates with a chiral
sterically hindered protonating agent. Thus, racemic
PhCH:NCHPhCO₂Me was deprotonated with LiN(CHMe₂)₂ and reprotonated
with **dipivaloyl-L-tartaric** acid to give 85%
L-PhCH:NCHPhCO₂Me.

AN 127:170461 CA
TI Chiral NMR shift reagents: mixtures of lanthanide tris(.beta.-diketonates) with chiral carboxylate anions
AU Wenzel, Thomas J.; Bean, Amy C.; Dunham, Sarah L.
CS Dep. Chemistry, Bates College, Lewiston, ME, 04240, USA
SO Magn. Reson. Chem. (1997), 35(6), 395-402
CODEN: MRCHEG; ISSN: 0749-1581
PB Wiley
DT Journal
LA English
AB Chiral carboxylic acids such as N-(R)-1-(1-naphthyl)ethylaminocarbonyl-L-tert-leucine, N-(R)-1-(1-naphthyl)ethylaminocarbonyl-L-valine and N-(3,5-dinitrobenzoyl)-L-leucine are solubilized in CHCl₃ by the addn. of NEt₃. The resulting ion pairs are useful **chiral resolving agents** for sulfoxides, amines and alcs. For certain substrates, particularly amines, the ion pairs were more effective **chiral resolving agents** than the corresponding org.-sol. ester derivs. Addn. of lanthanide tris(.beta.-diketonate) complexes to mixts. of the ion pairs causes enhancements in the enantiomeric resoln. in the spectra of certain substrates. The chiral carboxylate compd. bonds directly to the lanthanide tris(.beta.-diketonate) to form an anionic species. Shifts in the spectra of substrates and enhancements in enantiomeric resoln. in the presence of the anionic species appear to be dominated by bonding of the substrate to the carboxylate moiety of the lanthanide complex. These new reagents are therefore complementary to mixts. of lanthanide tris(.beta.-diketonate) complexes with the ester forms of the resolving agents.

L14 ANSWER 2 OF 2 CA COPYRIGHT 1998 ACS
AN 117:7401 CA
TI Lanthanide-**chiral resolving agent** mixtures as **chiral** NMR shift reagents
AU Wenzel, Thomas J.; Morin, Celeste A.; Brechting, Alice A.
CS Dep. Chem., Bates Coll., Lewiston, ME, 04240, USA
SO J. Org. Chem. (1992), 57(13), 3594-9
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
OS CJACS-IMAGE; CJACS
AB Mixts. of lanthanide complexes with sol. analogs of chiral liq. chromatog. stationary phases are shown to be useful NMR shift reagents for detg. enantiomeric excess. The chiral resoln. agents used in this work exhibit different assocn. consts. with enantiomeric substrates and assoc. weakly, if at all, with lanthanide ions. If the lanthanide assoc. with the substrate, the resoln. obsd. in the spectrum of the substrate is enhanced. Enhancement occurs because the enantiomer concd. in the bulk soln. spends more time bonded to the lanthanide ion than the enantiomer with a higher assocn. const. with the **chiral resolving agent**. Since the mechanism of interaction of many chiral liq. chromatog. phases is understood, or offers the potential to be understood, it should be possible to assign abs. configurations to the resolved NMR spectra. The method is applicable with donor-acceptor **chiral resolving**

agents such N-(3,5-dinitrobenzoyl)-L-leucine and
chiral hosts such as the cyclodextrins.

AN 113:152142 CA
TI Method of **resolving** cis 3-amino-4-(2-(2-furyl)eth-1-yl)-(1-methoxycarbonylmethyl)azetidin-2-one
IN Wright, Ian G.
PA Lilly, Eli, and Co., USA
SO U.S., 4 pp.
CODEN: USXXAM
PI US 4923983 A 900508
AI US 89-386664 890731
DT Patent
LA English

=> d abs

L18 ANSWER 1 OF 1 CA COPYRIGHT 1998 ACS
AB The title compd. (I) is **resolved**. cis-.alpha.,.alpha./.beta.,.beta.-I in MeCN was treated with L-(+)-**tartaric** acid dissolved in H2O and MeCN, heated to 35.degree. and seeded with L-(+)-**tartaric** acid salt of the cis-.beta.,.beta.-isomer. The mixt. was then allowed to cool overnight and filtered; 72.3% yield; 89.2% enantiomeric excess.